

QUALITY ASSURANCE PROJECT PLAN

for the

MINERGY CORPORATION

GLASS FURNACE TECHNOLOGY DEMONSTRATION

in

WINNECONNE, WISCONSIN

DEMONSTRATION PROGRAM

JUNE 2001

**U.S. Environmental Protection Agency
National Risk Management Research Laboratory
Cincinnati, Ohio**



QUALITY ASSURANCE PROJECT PLAN
MINERGY GLASS FURNACE TECHNOLOGY EVALUATION

Prepared for

U.S. ENVIRONMENTAL PROTECTION AGENCY
Office of Research and Development
National Risk Management Research Laboratory
Cincinnati, Ohio

Work Assignment Number	:	0-57
Date Prepared	:	June 25, 2001
Contract Number	:	68-C5-037
EPA Work Assignment Manager	:	Marta Richards
Telephone	:	513/569-7692
Prepared by	:	Tetra Tech EM, Inc.
Tetra Tech Project Manager	:	Joe Dauchy
Telephone	:	262/821-5894 ext. 235

QUALITY ASSURANCE PROJECT PLAN DISTRIBUTION LIST

Marta K. Richards	EPA SITE Project Manager SITE Point of Contact	(513) 569-7692
Jim Hahnenberg	EPA Region 5 Regional Project Manager	(312) 353-4213
Scott Cieniawski	EPA Great Lakes National Program Office Project Officer	(312) 353-9184
Robert Paulson	Wisconsin Department of Natural Resources Project Manager	(608) 266-7790
Joe Dauchy	Tetra Tech Project Manager Report Preparation	(262) 821-5894 ext. 235
Ken Partymiller	Tetra Tech Quality Control Coordinator	(502) 867-1397
Ken Brown	Tetra Tech Field Manager Field Demonstrations	(262) 821-5894 ext. 225
Gary Folk	General Electric Energy and Environmental Research Corp. Senior Analytical Chemist	(919) 460-1060
Terry Carroll	Minergy Corporation Point of Contact	(920) 727-1411
Tom Baudhuin	Minergy Corporation Manager of Engineering	(920) 727-1424
Greg Swanson	Tetra Tech QA Manager	(619) 718-9676 ext. 108

CONTENTS

<u>Section</u>	<u>Page</u>
1.0 PROJECT DESCRIPTION	1-1
1.1 Site Background	1-2
1.2 Previous Investigations	1-3
1.3 The Glass Furnace Technology	1-4
1.3.1 General Description of the Glass Furnace Technology	1-4
1.3.2 Minergy's Glass Furnace Technology	1-5
1.3.3 Site-Specific Configuration	1-7
1.4 Project Objectives	1-9
1.4.1 Primary Objectives	1-9
1.4.2 Secondary Objectives	1-15
1.5 Project Schedule	1-17
2.0 PROJECT ORGANIZATION AND RESPONSIBILITIES	2-1
2.1 Environmental Protection Agency National Risk Management Research Laboratory Superfund Innovative Technology Evaluation	2-1
2.2 Environmental Protection Agency Region 5	2-3
2.3 Wisconsin Department of Natural Resources	2-3
2.4 Minergy Corporation	2-3
2.5 Tetra Tech EM Incorporated	2-4
2.6 General Electric Energy and Environmental Research Corporation	2-5
2.7 Hazen Research, Incorporated	2-6
2.8 Laboratories	2-6
2.9 Project Organization for Quality Assurance	2-6
2.10 Personnel Locations	2-7
3.0 EVALUATION DESIGN	3-1
3.1 Operating Parameters and Sampling Strategy	3-1
3.1.1 Glass Furnace Technology System Installation and Operating Conditions	3-1
3.1.2 Sediment and Product Sampling to Meet Primary Objectives	3-2
3.1.2.1 Pilot Test Sampling	3-3
3.1.2.2 Glass Furnace Technology Treatment Sampling	3-6
3.1.3 Sediment and Product Sampling to Meet Secondary Objectives	3-8
3.2 Data Analysis and Presentation of Results	3-10

<u>Section</u>	<u>CONTENTS (Continued)</u>	<u>Page</u>
4.0	FIELD SAMPLING METHODOLOGIES AND PROCEDURES	4-1
4.1	Site Location	4-2
4.2	On-Site Facilities for the Field Sampling Team	4-2
4.2.1	Drying Process Testing Facilities	4-2
4.2.2	Glass Furnace Technology Process Testing Facilities	4-3
4.3	Sampling Locations	4-4
4.4	Sampling Events	4-12
4.5	Sediment Sampling Procedures	4-13
4.5.1	Pilot-Scale Dryer Sampling	4-13
4.5.2	Glass Furnace Technology Demonstration Sampling	4-14
4.6	Flue Gas Sampling Procedures	4-15
5.0	ANALYTICAL METHODS AND CALIBRATION PROCEDURES	5-1
5.1	Selection of Analytical Methods	5-1
5.2	Analytical Methods	5-2
5.3	Calibration Procedures	5-2
5.4	Sample Storage and Disposal	5-2
6.0	QUALITY ASSURANCE OBJECTIVES / QUALITY CONTROL REQUIREMENTS	6-1
6.1	Quality Assurance Objectives	6-1
6.1.1	Precision and Accuracy	6-1
6.1.2	Representativeness	6-5
6.1.3	Completeness	6-5
6.1.4	Comparability	6-5
6.1.5	Reporting Limits	6-6
6.2	Calculation of Data Quality Indicators	6-6
6.2.1	Precision	6-6
6.2.2	Accuracy	6-7
6.2.3	Representativeness	6-8
6.2.4	Completeness	6-8
6.3	Internal Quality Control Requirements	6-8
6.3.1	Field Quality Control Checks	6-9
6.3.1.1	Equipment Blanks and Field Blanks	6-9
6.3.1.2	Field Duplicate Samples	6-10
6.3.2	Laboratory Quality Control Checks	6-10
6.3.2.1	Method Blank Samples	6-10
6.3.2.2	Matrix Spike/Matrix Spike Duplicates	6-11
6.3.2.3	Laboratory Control Sample /Laboratory Control Sample Duplicates	6-11

7.0	FIELD AND LABORATORY DATA PROCESSING	7-1
7.1	Field Data Processing	7-1
7.2	Laboratory Data Processing	7-1
7.2.1	Laboratory Data Reduction	7-1
7.2.2	Laboratory Data Validation	7-2
7.2.3	Laboratory Reporting Requirements	7-3
7.3	Project Data Reporting	7-5
8.0	ASSESSMENTS	8-1
8.1	Quality Assurance Audits	8-1
8.1.1	Internal Audits	8-2
8.1.2	External Audits	8-4
8.2	Corrective Actions	8-4
8.3	Quality Assurance Reports to Management	8-6
9.0	REFERENCES	9-1

APPENDICES

Appendix A	Detailed Flue Gas Sampling Procedures
Appendix B	Analytical Methods
Appendix C	Calibration Procedures

FIGURES

<u>Figure</u>		<u>Page</u>
2-1	Project Organization	2-2
4-1	Sampling Locations for the Drying Process	4-6
4-2	Schematic of Glass Furnace Technology Process	4-7
7-1	Data Reporting Flowchart	7-4

TABLES

<u>Table</u>		<u>Page</u>
1-1	List of Polychlorinated Biphenyl Congeners of Concern	1-11
1-2	Criteria for Beneficial Reuse	1-13
3-1	Demonstration Samples to be Collected	3-4
4-1	Sampling Locations and Identifications During Drying Process	4-4
4-2	Key to Sampling Locations for Minergy Melting Operation	4-7
5-1	Analytical Methods for Drying Phase	5-3
5-2	Analytical Methods for Vitrification Phase	5-4
6-1	Quality Assurance Objectives for Critical Parameters	6-2

ACRONYMS AND ABBREVIATIONS

µg/L	microgram per liter
µg/m ³	micrograms per cubic meter
µm	micrometer
ASTM	American Society for Testing and Materials
BFB	bromofluorobenzene
Btu	British thermal units
CCV	continuing calibration verification
CEM	Continuous Emission Monitor
cfh	cubic feet per hour
cfm	cubic feet per minute
cm	centimeter
C	Celsius
COC	contaminant of concern
CRDL	contract-required detection limit
CVAA	cold vapor atomic absorption
DFTPP	decafluorotriphenylphosphine
EPA	U.S. Environmental Protection Agency
F	Fahrenheit
ft ²	square feet
ft ² /ton	square feet/ton
g	gram
GC/MS	gas chromatography/mass spectrometry
GE EER	General Electric Energy & Environmental Research Corporation
GFAA	graphite furnace atomic absorption
GFT	Glass Furnace Technology
GS	Greenberg-Smith
H ₂ O ₂	hydrogen peroxide
H ₂ SO ₄	sulfuric acid
Hazen	Hazen Research, Inc.
HCl/Cl ₂	hydrogen chloride/chlorine
HNO ₃	nitric acid
HRGC	high resolution gas chromatography
HRMS	high resolution mass spectrometry
IC	ion chromatography
ICP	inductively coupled plasma
ICAP	inductive coupled argon plasma
ITER	Innovative Technology Evaluation Report
Kemron	Kemron Environmental Services
KMnO ₄	potassium permanganate
L/min	liters per minute
LCS	laboratory control sample

ACRONYMS AND ABBREVIATIONS (continued)

LCSD	laboratory control sample duplicate
LRMS	low resolution mass spectrometry
m ³ /min	cubic meters per minute
MCAWW	“Methods for Chemical Analysis of Water and Wastes”
MDL	method detection limit
mg/kg	milligram per kilogram
Minergy	Minergy Corporation
mL	milliliter
mM	millimolar
mmHg	millimeter mercury
MS	matrix spike
MSD	matrix spike duplicate
m/z	mass-to-charge ratio
NA	not applicable
NaOH	sodium hydroxide
NIST	National Institute of Science and Technology
NRMRL	National Risk Management Research Laboratory
ORD	U.S.EPA Office of Research and Development
oz	ounce
PCB	polychlorinated biphenyl
PCDD	polychlorinated dibenzodioxin
PCDF	polychlorinated dibenzofuran
PFK	perfluorokerosene
POHC	principal organic hazardous compound
ppm	parts per million
QA	quality assurance
QAPP	Quality Assurance Project Plan
QC	quality control
RE	removal efficiency
RE _{GM}	geometric mean removal efficiency
RPD	relative percent difference
SITE	Superfund Innovative Technology Evaluation
SMU	Sediment Management Unit
SM-VOC	sampling method for volatile organic compounds
SVOC	semivolatile organic compound
TC	thermocouple
TCLP	toxicity characteristic leaching procedure
TE	Treatment efficiency
Tetra Tech	Tetra Tech EM Inc.
TSA	technical systems audit
UCB	upper confidence bound
VOC	volatile organic compound

ACRONYMS AND ABBREVIATIONS (continued)

VOST	volatile organic sampling train
WDNR	Wisconsin Department of Natural Resources
WHO	World Health Organization
XAD-2	porous polymeric resin

QUALITY ASSURANCE PROJECT PLAN

The U.S. Environmental Protection Agency (EPA) established the Superfund Innovative Technology Evaluation (SITE) Program to accelerate development, evaluation, and use of innovative technologies to remediate hazardous waste sites. The evaluation portion of the SITE Program focuses on technologies in the pilot- or full-scale stage of development. The evaluations are intended to collect performance data of known quality, and accordingly, sampling and analytical procedures are critical. Approved quality assurance and quality control (QA/QC) procedures must be stringently applied throughout the evaluation.

Tetra Tech EM Inc. (Tetra Tech) prepared this Quality Assurance Project Plan (QAPP) to follow the guidelines in the EPA National Risk Management Research Laboratory (NRMRL) requirements document entitled QAPP Requirements for Applied Research Projects, dated August 18, 1998. This QAPP describes how the SITE project team will collect and analyze samples to evaluate the Glass Furnace Technology developed by Minergy Corporation. Tetra Tech prepared this QAPP for EPA under Contract No.68-C5-037, Work Assignment No. 0-57.

1.0 PROJECT DESCRIPTION

Tetra Tech EM Inc. (Tetra Tech) prepared this Quality Assurance Project Plan (QAPP) for the U.S. Environmental Protection Agency (EPA) to provide guidance for evaluating the ability of the Glass Furnace Technology (GFT) to treat polychlorinated biphenyl (PCB) contamination from dredged-and-dewatered river sediment. This technology evaluation is being conducted by the EPA National Risk Management Research Laboratory (NRMRL) Superfund Innovative Technology Evaluation (SITE) Program in cooperation with the Wisconsin Department of Natural Resources (WDNR).

A GFT process has been developed and configured for this SITE demonstration by Minergy Corporation (the vendor). The process consists of two basic steps: a sediment drying step and the vitrification step. A full-scale unit will have these two process steps linked into a single unit to accomplish the sediment treatment. Because there is potential to lose sediment contaminants during the drying step and the potential for incomplete ($< 100\%$) contaminant treatment during the vitrification step, both processes will be evaluated. For the purposes of this pilot-scale evaluation, it is necessary to evaluate these two steps independently.

For the SITE demonstration, the sediment must contain no more than 10 percent moisture in order for the glass furnace to operate at optimal efficiency. Minergy has researched available sediment drying technology and has determined that, from an engineering standpoint, a Holoflite® dryer (dryer) unit is the most appropriate drying technology for the GFT treatment process. Due to the unavailability of large-scale Holoflite® dryers, a suitable bench-scale Holoflite® dryer located at the Hazen Research, Inc. (Hazen), facility in Golden, Colorado will be used to dry a representative amount of the sediment under very similar conditions to those in a large-scale dryer unit. The dryer unit is configured to allow sample collection of all waste and process streams, including off-gases. The vitrification phase of the process will be evaluated at a pilot-scale melter (melter) that is specifically designed for this evaluation and is currently under construction at Minergy's facility in Winneconne, Wisconsin. For clarification, this document refers to the Holoflite® dryer as the sediment dryer, and the pilot-scale melter portion of the GFT as the melter.

The sediment, glass aggregate, and waste streams will be analyzed for contaminants of concern (COCs) (1) before and after treatment in the bench-test sediment dryer and (2) before and after processing through the glass furnace. COCs include PCBs, dioxins and furans, metals including mercury, and semivolatile organic compounds (SVOCs).

This section provides information on the site background previous investigations, summarizes the GFT process, details the project objectives, and provides the project schedule. Section 2.0 describes the project organization and participant responsibilities. Section 3.0 provides details of EPA's evaluation design and statistical approach to evaluate the GFT. Section 4.0 describes EPA's field sampling program. Subsequent sections describe analytical methods and quality assurance and quality control (QA/QC) objectives and procedures.

1.1 SITE BACKGROUND

The GFT process is designed to treat PCB-contaminated sediment. The GFT project is being funded, in part, by a cooperative partnership between the Minergy, WDNR, and EPA's Great Lakes National Program Office (GLNPO), formed to evaluate potential treatment technologies for sediment throughout the state and Great Lakes region and is not intended to be specific to any one particular site.

Therefore, site-specific background data is not relevant, and detailed information regarding site location, geology, and hydrology is not necessary for this demonstration project. However, to conduct the evaluation of this treatment technology, river sediment dredged from the Lower Fox River will be used.

The sediment to be used in this evaluation was obtained from the Lower Fox River during the 1999 Sediment Management Unit (SMU) 56/57 pilot dredging project, which included hydraulic dredging, on-shore dewatering, filter pressing, treatment with lime, and transporting and disposing of PCB-containing sediment. WDNR conducted oversight on the project with funding from the Fox River Group. The Fox River Group includes seven paper manufacturing companies responsible for the PCB contamination that participated in the design and implementation of the dredging project. The SMU

56/57 project goal was to “generate information to assess the effectiveness and expense of large-scale sediment dredging and disposal of contaminated sediment from the Lower Fox River” (WDNR 1999).

The SMU 56/57 dredging project, from which the sediment for the GFT evaluation was obtained, ran from August to December 1999. In general, the dredging project consisted of hydraulic dredging a portion of the river bottom into two lined settling basins. After the solids settled out, they were pumped to plate and frame presses for mechanical dewatering. Lime was added, on an as-needed basis, to aid solidification, and the sediment was transported to a local landfill for disposal. Water was treated with sand filtration and activated carbon before being discharged back into the Lower Fox River.

A portion of the sediment from the SMU 56/57 project was segregated for the purpose of evaluating innovative sediment treatment technology. On December 17, 1999, rather than loading dredged-and-dewatered sediment into trucks for transport and disposal, it was loaded into four, 20-cubic-yard, lined roll-off boxes. The boxes were covered with tarps and transported to the Brown County East Landfill in Green Bay, Wisconsin, where they were temporarily stored until needed for the GFT evaluation.

1.2 PREVIOUS INVESTIGATIONS

The Lower Fox River sediment has been subjected to various studies over the last 15 years. Sediment in the vicinity of SMU 56/57 consists of 60 to 80 percent silt with lesser amounts (0 to 40 percent) of sand and clay. PCB concentrations as high as 710 parts per million (ppm) have been detected in samples collected from SMU 56/57. However, analytical results for the sediment stockpiles prior to and immediately following the sediment acquisition for the GFT evaluation, indicated PCB concentrations less than 50 ppm. The stockpiled sediment, which was filter-pressed, was also characterized as being approximately 50% solids, and as having an average mercury concentration of approximately 1 ppm.

1.3 THE GLASS FURNACE TECHNOLOGY

The following sections provide a general description of the GFT, as well as Minergy's melter and its site-specific configuration.

1.3.1 General Description of the Glass Furnace Technology

Glass furnaces have been used for decades in industrial glass manufacturing. The process design of a glass furnace is focused on melting low energy feedstock, that is, materials with low energy content as measured in British thermal units (Btu). Feedstock consisting primarily of silica sand melts in the furnace, and the molten product is cooled to form glass. Silica is one of the primary constituents of river sediment, and in this case, the glass furnace technology utilizes vitrification of river sediment with the expectation of destroying contaminants of concern and creating a useable aggregate as a final product. Other thermal destruction processes are too costly to be appropriate for use on river sediment because the sediment have very little fuel value. Other processes rely on the significant organic content (fuel content) of the feed material, but because very little energy is contained in sediment, large quantities of auxiliary fuel or of electric power would have to be used.

Minergy and WDNR have successfully completed two phases of a multiphase feasibility study to evaluate GFT as a remediation alternative. The first phase (Phase I) involved characterizing the mineral composition of river sediment to estimate the glass quality, durability, and melting points. Data gathered during Phase I indicated that the characteristics of river sediment are consistent throughout the river and are favorable for producing a quality glass product. Based on mineral composition, combustibility, moisture content, and costs to operate, Minergy claims that analysis of the sediment indicates vitrification technology is more appropriate for river sediment than incineration.

In the second phase of the project (Phase II), sediment from the Lower Fox River was melted in a crucible to determine the glass characteristics and the qualities of the sediment, both alone and when

augmented with other materials (flux mixtures). Four different test “recipes” were included in the crucible melts, and the sediment was successfully melted into glass in all four tests. Data obtained during the second phase were used to develop (1) a proposed “recipe” for melting river sediment into glass aggregate and (2) preliminary engineering designs for the pilot-scale facility proposed for the third phase. The preliminary engineering analysis indicated that it was not practical or cost-efficient to use an existing glass furnace for GFT testing. This analysis indicated that it would cost as much to retrofit an existing facility to the specifications needed to melt the sediment as it would to build a pilot glass furnace to the same specifications. Also, it was discovered that most existing manufacturing facilities are too large to accommodate a limited duration test.

Results of the feasibility study also indicated that the capital and operating costs of the GFT provide for an economically viable option for treating contaminated river sediment.

1.3.2 Minergy’s Glass Furnace Technology

The first step of Minergy’s GFT process includes the handling of dewatered sediment from the dredging site. Sediment is delivered in dewatered form, in the range of 45 to 55 percent solids (by weight).

The next step of the process includes partially drying the sediment. Drying the sediment limits the amount of moisture in the melter, thereby reducing the physical volume of the feed and maintaining high processing temperatures. Gases from the drying step are directed into the glass furnace or into another destruction device to control emissions of contaminants of concern. Several technologies are available for thermal drying. Suggested technologies include those that use indirect drying systems to reduce the volume of gas requiring treatment. The Holoflite® dryer evaluated in this EPA SITE Program is one such system. Equipment selected for use on river sediment will depend on the characteristics of the sediment and the overall thermal cycle of the project.

In the next step of the process, the sediment passes from the drying system into a glass furnace. The

glass furnace is a refractory-lined, rectangular melter. The refractory is brick or concrete that has been specially treated to resist chemical and physical abrasion, has a high melting point, and provides a high degree of insulating value to the process. Natural gas is fired in the furnace, raising the internal temperatures to about 3,000° Fahrenheit (F). At this temperature, the sediment melts and flows out of the furnace as molten glass.

The molten material is then collected and quickly cooled in a water quench system to form the glass aggregate product. Minergy claims that glass aggregate can be stored and handled similarly to conventional quarried aggregates. Some off-site crushing and screening would be required to meet the particle size specifications of particular aggregate markets.

The high temperature environment in the melter is expected to completely destroy any organic compounds that may be contained in the sediment. In addition, trace metals in the sediment are expected to be stabilized in the glass aggregate product, and are anticipated to be biologically and chemically inert. Minergy claims off-gas treatment is simplified and energy efficiency improved by the melter's use of purified oxygen rather than atmospheric air as the oxygen source.

Minergy has intellectual property protection for the application of GFT on contaminated sediment. Minergy has made modifications to the standard glass furnace design which have been incorporated to best suit this application, including the following:

- The use of a water quench system to quickly harden the molten glass and increase the inert characteristics of the final product. Glass melters typically use annealing or other slow-cooling processes to enhance glass clarity and other product qualities. These product qualities are not applicable to the manufacture of glass aggregate because of its intended final use as a construction product.
- The use of a "shallow" glass pool inside the melter. Glass melters typically have deeper pools of glass inside the melter, taking advantage of the low opacity of the glass being produced. Molten sediment is quite opaque, thus reducing energy transfer by radiation.

- The use of refractory selected by evaluating the abrasive qualities of the molten sediment and a heat flow analysis for each construction type.
- The use of flux materials that have been selected based on the high temperature viscosity of the molten sediment material.

1.3.3 Site-Specific Furnace Configuration

The pilot-scale glass furnace, or melter, is designed to simulate a full-scale production unit for the generation of glass aggregate from sediment. To produce an adequate simulation, some assumptions have been made regarding the full-scale melter based on typical glass-manufacturing practices. The pilot-scale melter is a scaled model of the full-scale melter and has been designed to operate in a similar manner.

Melter characteristics are given in the box below:

Pilot-Scale Melter Characteristics

Aspect Ratio	2:1
Area	10 square feet (ft ²)
Melting Rate	5.4 square feet per ton (ft ² /ton)
Dwell Time	6 hours
Gas Usage	1.7 MM Btu/hour
Oxygen Usage	35 cubic feet per hour (cfh)
MM Btu/ton	20.9 MM Btu/ton
Output	2 tons/day

The pilot-scale melter area is 10 square feet with a 2:1 aspect ratio. The melter is fired with oxygen and natural gas to utilize the best available control technology for nitrogen-related emissions and particulate matter. The melter will have eight split-stream oxy-fuel burners to approximate the eight burners used

in a full-scale melter. The glass quality is expected to be adequate for use as aggregate after a 6-hour processing time at a shallow glass level.

The materials selected are typical for soda-lime glass operations in an oxy-fuel environment. Six inches have been added to the height of the glass processing area to provide additional volume for destruction of organics contained in the sediment feedstock. The flue is located in the front of the melter, which is not the traditional location for oxy-fuel furnaces. However, this configuration allows any fine particulate that become entrained in the exhaust gases to have the maximum time in the furnace allowing these particulates to be melted, or minimized.

The glass will flow under a skimmer block into the forehearth. The forehearth has been constructed in a conventional manner, with the glass outlet flowing to the water quench system. This method is used in other frit-making operations.

The batch charger is a standard screw system that has been used universally in glass furnaces. The screw charger was chosen for its ability to tightly seal the hopper to the charger and the charger to the furnace. The tight seals minimize dust formation in the raw material feedstock. The charger is similar in size to those used in a full-scale unit. It has been retrofitted with a small screw barrel and flights for the pilot-scale melter. The charger can be reused within a full-scale melter by modifying the barrel and flights.

Both the gas and oxygen skids have essentially the same safety system. A strainer is used prior to a pressure regulator, and a high-low pressure switch is tied to the double block automatic shut-off valves. A differential pressure switch is used to determine flow through the system to safeguard against injecting raw natural gas or oxygen into the furnace. If either natural gas or oxygen flow is lost, the skid shuts down that zone. Each zone is then automatically regulated for gas and oxygen flows by a signal from the mass flow meter to a process control loop back to an automatic valve.

The pilot-scale melter is regulated by process control loops to the melter and forehearth. The control loops use thermocouple signals to maintain a constant temperature and automatically adjust the gas and oxygen for each zone. The control panel contains three single loop controllers, three digital gas flow meters, three digital oxygen flow meters, six digital temperature meters, status lights for the main fuel train, E-stop, alarm horn, and alarm silence push button.

Refractory brick was selected for the pilot-scale melter based evaluating the abrasive qualities of the molten sediment and an analysis of heat flow for each construction type. The analyses were conducted to ensure that the materials should not be placed in temperatures beyond their capability and to determine the total heat loss of the entire system.

1.4 PROJECT OBJECTIVES

The technology evaluation has both primary and secondary objectives. Primary objectives are considered critical for the technology evaluation. Secondary objectives provide additional information that is useful, but not critical. To obtain the data required to meet the specified primary project objectives, EPA will collect and analyze sediment samples before the test, after the drying processes are complete, and after the melter, when the sediment will have been converted into glass aggregate product. The sediment and glass aggregate samples will be analyzed for COC concentrations, and the results will provide data on pre- and post-drying and pre- and post-melter COC concentrations. These data will also be used to determine the PCB treatment efficiency of the Minergy GFT. Details of the experimental approach and the procedures involved in analyzing the data are given in Section 3.0.

1.4.1 Primary Objectives

The following primary objectives are considered critical to the success of the evaluation. For each objective, a brief description of the experimental approach is given.

P1 To determine the treatment efficiency (TE) of PCBs in dredged-and-dewatered river sediment when processed in the Minergy GFT.

The concentration of PCBs in the river sediment will be determined by analyzing composite samples of the dredged-and-dewatered sediment (sediment) collected prior to treatment. Flue gas samples will be collected using EPA Method 23 (40 CFR Part 60, Appendix A). Quench water samples and aggregate product samples will be collected as composite samples. The PCB content of the flue gas, quench water, condensate, sediment, and aggregate product will be determined using Method 680. Table 1-1 lists the congeners of concern to be analyzed by Method 680. Selected samples will also be analyzed for dioxins and furans (Method 8290), PCBs (crushed aggregate will be analyzed by both Method 1668 and 680), VOCs (Method 8260B), SVOCs (Method 8270C), and metals including mercury (Methods 6010B/7470A/7471A).

The TE of the GFT process will be calculated as follows:

$$TE = 100\% \times (W_{in} - W_{out}) / W_{in}$$

Where:

W_{in} = Mass of PCBs entering the system:
For the overall GFT process (drying and melting), W_{in} represents the PCB concentration of the dewatered sediment; for the melting system only, W_{in} represents the PCB concentration of the dried sediment

Section: 1.0
Revision: 2
Date: 7/24/01
Page: 1-11

Table 1-1 List of PCB Congeners of Concern

CASRN	Current BZ and IUPAC Number	Name
2051-60-7	BZ 1	2-ChloroB
37680-73-2	BZ 101/90	2,2',4,5,5'-PentachloroB
32598-14-4	BZ 105	2,3,3',4,4'-PentachloroB
70424-68-9	BZ 107	2,3,3',4',5-PentachloroB
38380-03-9	BZ 110/77	2,3,3',4',6-PentachloroB
74472-37-0	BZ 114	2,3,4,4',5-PentachloroB
31508-00-6	BZ 118	2,3',4,4',5-PentachloroB
56558-17-9	BZ 119	2,3',4,4',6-PentachloroB
65510-44-3	BZ 123	2,3',4,4',5'-PentachloroB
57465-28-8	BZ 126	3,3',4,4',5-PentachloroB
38380-07-3	BZ 128	2,2',3,3',4,4'-HexachloroB
38380-05-1	BZ 132/168	2,2',3,3',4,6'-HexachloroB
52744-13-5	BZ 135/144	2,2',3,3',4,6'-HexachloroB
38411-22-2	BZ 136	2,2',3,3',6,6'-HexachloroB
35694-06-5	BZ 137	2,2',3,4,4',5-HexachloroB
35065-28-2	BZ 138/163	2,2',3,4,4',5'-HexachloroB
52712-04-6	BZ 141	2,2',3,4,5,5'-HexachloroB
51908-16-8	BZ 146	2,2',3,4',5,5'-HexachloroB
38380-04-0	BZ 149/123	2,2',3,4',5',6-HexachloroB
52663-63-5	BZ 151	2,2',3,5,5',6-HexachloroB
35065-27-1	BZ 153/184	2,2',4,4',5,5'-HexachloroB
38380-08-4	BZ 156/171	2,3,3',4,4',5-HexachloroB
69782-90-7	BZ 157	2,3,3',4,4',5'-HexachloroB
74472-42-7	BZ 158	2,3,3',4,4',6-HexachloroB
38444-78-9	BZ 16/32	2,2',3-TrichloroB
52663-72-6	BZ 167	2,3',4,4',5,5'-HexachloroB
32774-16-6	BZ 169	3,3',4,4',5,5'-HexachloroB
37680-66-3	BZ 17	2,2',4-TrichloroB
35065-30-6	BZ 170	2,2',3,3',4,4',5-HeptachloroB
52663-74-8	BZ 172	2,2',3,3',4,5,5'-HeptachloroB
38411-25-5	BZ 174	2,2',3,3',4,5,6'-HeptachloroB
52663-65-7	BZ 176	2,2',3,3',4,6,6'-HeptachloroB
52663-70-4	BZ 177	2,2',3,3',4,5',6'-HeptachloroB
52663-67-9	BZ 178/126	2,2',3,3',5,5',6-HeptachloroB
37680-65-2	BZ 18	2,2',5-TrichloroB
35065-29-3	BZ 180	2,2',3,4,4',5,5'-HeptachloroB
52663-69-1	BZ 183	2,2',3,4,4',5',6-HeptachloroB
52712-05-7	BZ 185	2,2',3,4,5,5',6-HeptachloroB
52663-68-0	BZ 187/182	2,2',3,4',5,5',6-HeptachloroB
39635-31-9	BZ 189	2,3,3',4,4',5,5'-HeptachloroB
38444-73-4	BZ 19	2,2',6-TrichloroB
41411-64-7	BZ 190	2,3,3',4,4',5,6-HeptachloroB

Notes: B - biphenyl
BZ - Ballschmitter and Zell Numbers
CASRN - Chemical Abstract Service Registry Number
IUPAC - International Union of Pure and Applied Chemistry

Section: 1.0
Revision: 2
Date: 7/24/01
Page: 1-12

Table 1-1 (Continued) List of PCB Congeners of Concern

CASRN	Current BZ and IUPAC Number	Name
2136-99-4	BZ 202	2,2',3,3',5,5',6,6'-OctachloroB
52663-76-0	BZ 203/196	2,2',3,4,4',5,5',6,-OctachloroB
40186-72-9	BZ 206	2,2',3,3',4,4',5,5',6-NonachloroB
52663-77-1	BZ 208	2,2',3,3',4,5,5',6,6'-NonachloroB
2051-24-3	BZ 209	DecachloroB
38444-85-8	BZ 22	2,3,4'-TrichloroB
55702-45-9	BZ 24	2,3,6-TrichloroB
55712-37-3	BZ 25	2,3',4-TrichloroB
38444-81-4	BZ 26	2,3',5-TrichloroB
38444-76-7	BZ 27	2,3',6-TrichloroB
7012-37-5	BZ 28	2,4,4'-TrichloroB
15862-07-4	BZ 29	2,4,5-TrichloroB
16606-02-3	BZ 31	2,4',5-TrichloroB
38444-86-9	BZ 33	2,4',6-TrichloroB
38444-90-5	BZ 37/42	3,4,4'-TrichloroB
13029-08-8	BZ 4	2,2'-DichloroB
38444-93-8	BZ 40	2,2',3,3'-TetrachloroB
41464-39-5	BZ 44	2,2',3,5'-TetrachloroB
70362-45-7	BZ 45	2,2',3,6-TetrachloroB
41464-47-5	BZ 46	2,2',3,6'-TetrachloroB
2437-79-8	BZ 47	2,2',4,4'-TetrachloroB
70362-47-9	BZ 48	2,2',4,5-TetrachloroB
41464-40-8	BZ 49	2,2',4,5'-TetrachloroB
16605-91-7	BZ 5	2,3-DichloroB
35693-99-3	BZ 52	2,2',5,5'-TetrachloroB
41464-41-9	BZ 53	2,2',5,6'-TetrachloroB
41464-43-1	BZ 56	2,3,3',4'-TetrachloroB
74472-33-6	BZ 59	2,3,3',6-TetrachloroB
25569-80-6	BZ 6	2,3'-DichloroB
33052-41-1	BZ 60/92	2,3,4,4'-TetrachloroB
74472-34-7	BZ 63	2,3,4',5-TetrachloroB
52663-58-8	BZ 64/41	2,3,4',6-TetrachloroB
32598-10-0	BZ 66/95	2,3',4,4'-TetrachloroB
332284-50-3	BZ 7	2,4-DichloroB
32598-11-1	BZ 70	2,3',4',5-TetrachloroB
41464-46-4	BZ 71	2,3',4',6-TetrachloroB
32690-93-0	BZ 74	2,4,4',5-TetrachloroB
70362-48-0	BZ 76	2,3',4',5'-TetrachloroB
32598-13-3	BZ 77	3,3',4,4'-TetrachloroB
34883-43-7	BZ 8	2,4'-DichloroB
70362-50-4	BZ 81	3,4,4',5-TetrachloroB
52663-62-4	BZ 82	2,2',3,3',4-PentachloroB
52663-60-2	BZ 84	2,2',3,3',6-PentachloroB
65510-45-4	BZ 85	2,2',3,4,4'-PentachloroB
38380-02-8	BZ 87/81	2,2',3,4,5'-PentachloroB
68194-05-8	BZ 91	2,2',3,4',6-PentachloroB
41464-51-1	BZ 97	2,2',3,4',5'-PentachloroB
38380-01-7	BZ 99	2,2',4,4',5-PentachloroB

Notes: B - biphenyl
BZ - Ballschmitter and Zell Numbers
CASRN - Chemical Abstract Service Registry Number
IUPAC - International Union of Pure and Applied Chemistry

W_{out} = Mass of PCBs leaving the system:
For the drying system only, W_{out} represents the PCB concentration of the dried sediment; for the overall GFT system, W_{out} represents the combined PCB concentrations of the process flue gas stream, the quench water stream, and the aggregate product.

P2 To determine whether the GFT glass aggregate product meets the criteria for beneficial reuse under relevant federal and state regulations. The aggregate product will be judged to be beneficial with respect to each metal or PCB if the 95 percent upper confidence bound (UCB) for the estimated mean (of each metal or PCB) is less than the federal or state regulatory requirements, as applicable.

The final glass aggregate product (aggregate) (D) from the GFT system will be subjected to a suitable aqueous extraction, such as the Synthetic Precipitate Leaching Procedure (SPLP), Method 1312 and the Shake Extraction of Solid Waste with Water, American Society of Testing and Materials (ASTM) Method D3987-85. The aqueous extraction procedure will be followed by EPA Method 6010B for metals, Method 7470A for Mercury, and Method 680 for PCBs. The results of these tests will be evaluated against federal and state requirements to determine if the aggregate exceeds SPLP leaching limits and is suitable for beneficial reuse.

For example, the WDNR will compare the results of these tests against their criteria for beneficial reuse and PCB concentration as listed in Table 1-2. Metal concentration criteria are from WDNR's statute on beneficial reuse and PCB criteria from WDNR's water quality standard.

Table 1-2
Criteria for Beneficial Reuse

Contaminant	Criteria for Water Leach Test
Arsenic	0.005 mg/L
Barium	0.4 mg/L
Cadmium	0.0005 mg/L
Chromium	0.010 mg/L
Lead	0.0015 mg/L
Mercury	0.0002 mg/L
Selenium	0.010 mg/L
Silver	0.010 mg/L
Total PCBs*	0.03 µg/L

* The criteria for PCBs were taken from WDNR regulation NR 140 (Table 1) Groundwater Quality Standards, while all others were taken from WDNR regulation NR 538 Beneficial Reuse of Industrial Byproducts.

An aqueous extraction will also be performed on a split sample of the aggregate product that is crushed and passed through a #200 sieve. The purpose of the crushing is to simulate the use of the aggregate product as road base material. In addition, the crushed aggregate sample will be analyzed for PCBs by Method 1668 (a high resolution gas chromatography/high resolution mass spectrometry [HRGC/HRMS] method) for a selected group of congeners, representing those congeners detected in the highest concentration in the dewatered sediment samples.

1.4.2 Secondary Objectives

The following secondary objectives are intended to provide additional information that will be useful in evaluating the technology.

S1 Determine the unit cost of operating the GFT on dredged-and-dewatered river sediment.

To determine the unit cost of removing the PCBs and the other organic and inorganic contaminants from the river sediment, the capital and operating cost data associated with both the dryer and the furnace will be collected from Minergy and assimilated to estimate unit costs for the pilot-scale test facilities. Minergy will work with EPA to estimate the equivalent costs for production-sized dryers and melters. Attendant air pollution control devices or wastewater treatment systems required for the units to maintain full compliance with federal and state emissions or effluent discharge limitations will be included in the capital and variable cost estimates. Specifically, this objective will be achieved by assessing the following expense categories:

- Mobilization
- Site preparation
- Permitting and regulatory compliance
- Capital equipment/equipment lease
- Startup
- Labor
- Consumable material
- Utilities
- GFT system implementability
- Waste disposal
- Residual waste shipping and handling

- Analytical services
- Demobilization

S2 Quantify the organic and inorganic contaminant losses resulting from the existing or alternative drying process used for the dredged-and-dewatered river sediment.

EPA will quantify the organic and inorganic material content of the dredged-and-dewatered sediment before and after the drying process. In this objective, concentrations of SVOCs, PCBs, metals, mercury, and dioxins and furans in the dredged-and-dewatered river sediment will be determined and compared to the same parameters in the dried river sediment.

This objective will be achieved by analyzing the river sediment, before and after drying; for SVOCs by Method 8270C; metals by Method 6010B; mercury by Method 7471A; PCBs by Method 680; and dioxins and furans by Method 8290.

S3 Characterize organic and inorganic constituents in all GFT process input and output streams.

EPA will analyze all input and output streams for organic and inorganic material content. In this objective, concentrations of volatile organic compounds (VOC), SVOCs, PCBs, metals, mercury, and dioxins and furans in the dried river sediment will be determined and compared to the same parameters in the effluent waste and product streams (VOCs in flue gas stream only).

This objective will be achieved by analyzing the dried river sediment for VOCs by Method 8260B; SVOCs by Method 8270C; metals by Method 6010B; mercury by Method 7471A; PCBs by Method 680; and dioxins and furans by Method 8290.

Flue gas stream samples will be collected using Method 0010 for VOCs and SVOCs; Method 0060 for metals and mercury; and Method 23 for PCBs, dioxins, and furans. VOCs will be analyzed by Method 8260B; SVOCs will be analyzed by Method 8270C; Metals will be analyzed by Method 6010B; mercury by Method 7470A; PCBs by Method 680; and dioxins and furans by Method 8290.

The quench water will be analyzed for SVOCs by Method 8270C; metals by Method 6010B; mercury by Method 7470A; PCBs by Method 680; and dioxins and furans by Method 8290.

The aggregate will be analyzed as the frit product from the melter and crushed (less than 200 mesh). SVOCs will be analyzed by Method 8270C; metals by Method 6010B; mercury by Method 7471A; PCBs by Method 680 and 1668 (crushed aggregate only); and dioxins and furans by Method 8290.

1.5 PROJECT SCHEDULE

The key steps in the technology evaluation are tentatively scheduled as follows:

Pilot-scale dryer test at Hazen facility in Golden, Colorado	January 2001
Sediment dried in preparation for melter test at Minergy facility	March 2001
Pilot-scale melter construction	April 2001
QAPP submitted for EPA review	May 2001
Melter test and sampling at Minergy facility	June 2001
Receipt of analytical data from laboratories	August 2001
Draft Innovative Technology Evaluation Report (ITER)	August 2001
Submit ITER for EPA review	September 2001

This schedule is subject change.

2.0 PROJECT ORGANIZATION AND RESPONSIBILITIES

The success of this evaluation project depends on a cooperative effort involving government and private parties. This section identifies these parties and their roles in the project. Figure 2-1 presents the project technical organization and assignments, as well as primary communication channels. Personnel responsibilities are described for EPA SITE, EPA Region 5, EPA's GLNPO, the WDNR, Minergy, Tetra Tech, General Electric Energy and Environmental Research Corporation (GE EER), Kemron Environmental Services (Kemron), and Paradigm Laboratories, as well as for the EPA SITE/Tetra Tech QA function. An organizational flow chart is presented as Figure 2-1.

2.1 EPA NATIONAL RISK MANAGEMENT RESEARCH LABORATORY SITE

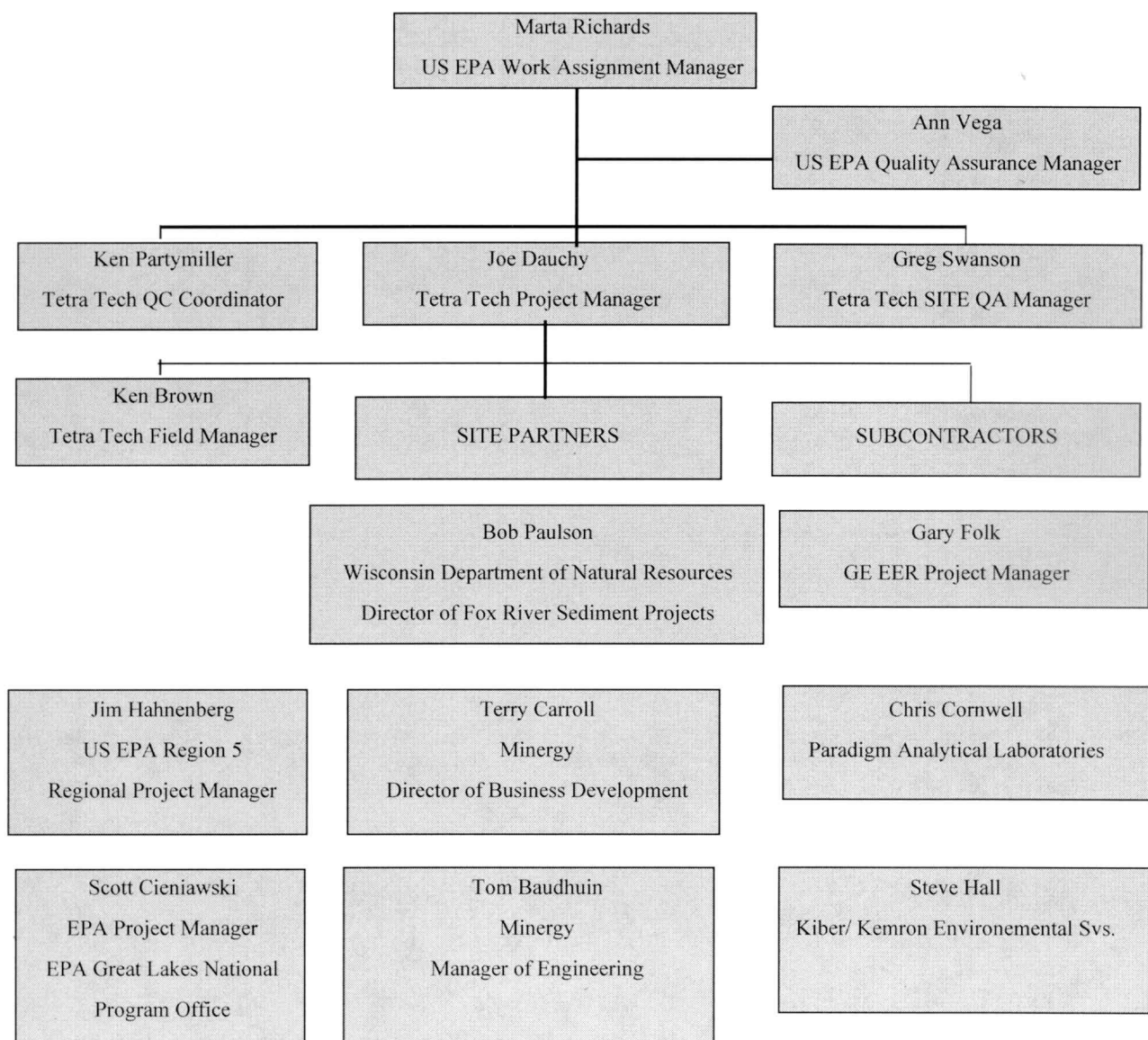
The EPA SITE Project Manager, Ms. Marta Richards, has overall responsibility for the evaluation project. However, EPA SITE has delegated to Tetra Tech the responsibility for comprehensive technical support to the technology evaluation (see Section 2.5).

The responsibilities of the EPA SITE Project Manager for the evaluation include the following:

- C Provide technical direction to EPA SITE's technical support contractor, Tetra Tech, during all phases of the project
- C Plan and coordinate meetings and communications among the various parties involved in this evaluation
- C Review and comment on Minergy's GFT demonstration procedures
- C Review and audit field activities
- C Ensure that technology evaluation requirements are met
- C Review and approve the QAPP and all final reports

Section: 2.0
Revision: 2
Date: 7/23/01
Page: 2-2

FIGURE 2-1 PROJECT ORGANIZATION
US EPA SITE DEMONSTRATION PROGRAM
MINERGY MELTER TECHNOLOGY DEMONSTRATION



2.2 EPA REGION 5

As the representative for the regional federal regulatory agency for this site, Mr. Jim Hahnenberg, EPA Region 5 Regional Project Manager, will be responsible for reviewing this QAPP and all final reports.

Scott Cieniawski is the representative for EPA's GLNPO and will review this QAPP and all final reports.

2.3 WISCONSIN DEPARTMENT OF NATURAL RESOURCES

Mr. Robert Paulson will represent the WDNR and function as a resource for information on the dredging operations and properties of the river sediment used in this demonstration. Mr. Paulson maintains the WDNR database of sediment data for the Fox River cleanup and evaluated several remedial alternatives to address the contaminant exposure pathways identified. Much of the work performed for the Fox River cleanup project has been under the direction of the WDNR.

2.4 MINERGY CORPORATION

The Minergy project manager, Terry Carroll, is responsible for supervising the sediment dryer and pilot-scale melter operations in accordance with the requirements outlined in this QAPP. Mr. Carroll will coordinate evaluation activities with Hazen, Tetra Tech, and EPA SITE to ensure that all requirements are met. Under Mr. Carroll's direction, Minergy will be responsible for the following:

- Drying samples of the sediment in a bench-scale dryer to allow for the representative sampling of in-going and out-going sediment, off-gasses, and condensed water to characterize any losses or transformations of COCs.
- Drying the sediment feedstock in a large-scale dryer to prepare it for the melter
- Construction, operation, and demobilization of the melter system

- Collecting system operating and performance data and transmitting these data to EPA
- Reviewing the QAPP and all final reports

2.5 TETRA TECH EM INC.

EPA SITE has contracted with Tetra Tech to provide comprehensive technical support for this evaluation project. The Tetra Tech project manager, Dr. Joseph Dauchy, is responsible for all tasks performed by Tetra Tech and for direct communication with evaluation participants. Dr. Dauchy is responsible for ensuring that all sampling, analytical, and QA/QC requirements are met for the project. Dr. Dauchy will prepare technical documents and coordinate technical communications with EPA SITE, WDNR, and Minergy. In addition, Dr. Dauchy will review sampling and analytical data obtained during the evaluation and will be responsible for preparing the final reports.

Dr. Dauchy's specific responsibilities as Tetra Tech project manager include the following:

- C Communicate with and receive technical direction from the EPA SITE Project Manager
- C Develop the QAPP, ITER, and other project deliverables
- C Manage staff
- C Provide required planning, cost, and schedule control
- C Maintain project file and written records documentation

Dr. Kenneth Partymiller is Tetra Tech's QC coordinator for this project. Dr. Partymiller will coordinate QC technical operations among project staff. Dr. Partymiller's specific responsibilities include the following:

- C Provide assistance and guidance in developing and revising the QAPP

- C Provide guidance and coordination to rapidly resolve QC problems
- C Review the quality of all project documentation, including data packages and reports

Tetra Tech's field manager, Mr. Ken Brown, will directly perform or will oversee all of Tetra Tech's field activities at the site. Mr. Brown will also serve as the acting project manager in the absence or unavailability of Dr. Dauchy. As field manager, Mr. Brown's specific responsibilities include the following:

- C Coordinate and schedule all field activities
- C Collect samples and perform field measurements in accordance with the QAPP
- C Oversee the activities of Tetra Tech and of Tetra Tech subcontractor personnel in the field
- C Comply with the health and safety plan HASP and oversee site health and safety activities during the technology demonstration
- C Ensure that treatment residuals and any hazardous waste produced during the demonstration are disposed of in compliance with federal, state, and local requirements

2.6 GENERAL ELECTRIC ENERGY AND ENVIRONMENTAL RESEARCH CORPORATION

GE EER has the responsibility to conduct stack sampling and measurement activities associated with this evaluation. The GE EER project manager, Mr. Gary Folk, is responsible for supervising the emissions sampling personnel and operations in accordance with the requirements outlined in this QAPP.

2.7 HAZEN RESEARCH, INC.

Hazen will operate and maintain the pilot-scale sediment dryer to determine the suitability of commercial-scale dryers of similar type for inclusion in the full-scale construction of the GFT system. The Hazen project manager, Mr. Dennis Johnson, is responsible for operation of the pilot test and assisting Tetra Tech in the collection of samples in accordance with the requirements of this QAPP.

2.8 LABORATORIES

Paradigm Analytical Laboratories of Wilmington, North Carolina, will perform analyses for PCBs and dioxins/furans on all media (sediment, water, and air). The Paradigm project manager, Mr. Chris Cornwell, is responsible for overseeing the analysis of samples submitted by Tetra Tech, reviewing the data for quality, and reporting results.

Kemron Environmental Services of Marietta, Ohio, will perform analyses for VOCs, SVOCs, and metals including mercury, on all media (sediment, water, and air). The Kemron project manager, Mr. Steve Hall, is responsible for overseeing the analysis of samples submitted by Tetra Tech, reviewing the data for quality, and reporting results.

2.9 PROJECT ORGANIZATION FOR QUALITY ASSURANCE

Because of the visibility and importance of the SITE program and the corresponding need to obtain data of known and documented quality, EPA SITE emphasizes QA for all SITE projects. Tetra Tech, as a prime contractor to EPA/SITE, is responsive to the QA requirements of EPA SITE and has instituted a parallel organization designed to achieve EPA SITE goals.

The EPA NRMRL Divisional Quality Assurance Manager, Ann Vega, is responsible for overseeing and reviewing project QA activities in support of the EPA SITE Project Manager. She is also responsible for communicating EPA NRMRL QA policy and guidance to Tetra Tech through the Tetra Tech SITE QA manager, Dr. Greg Swanson. Ms. Vega and Dr. Swanson have parallel responsibilities within their respective organizations. These responsibilities include the following:

- Review and comment on the QAPP and all final reports
- Set policy and provide guidance for the conduct of field and laboratory audits; provide oversight for all field and laboratory audits
- Provide general QA guidance and consultation on an as-needed basis throughout this evaluation

2.10 PERSONNEL LOCATIONS

The locations and telephone numbers of the Minergy technology evaluation participants are as follows:

- Ms. Marta Richards and Ms. Ann Vega
U.S. Environmental Protection Agency
Office of Research and Development
26 W. Martin Luther King Drive
Cincinnati, Ohio 45268
Phone: 513/569-7692 and 513/569-7635
Fax: 513/569-7676 and 513/569-7585
Email: richards.marta@epa.gov
Email: vega.ann@epa.gov
- Mr. Robert Paulson
Wisconsin Department of Natural Resources
Bureau of Watershed Management
101 S. Webster Street, P.O. Box 7921
Madison, Wisconsin 53707-7921
Phone: 608/266-7790
Fax: 608/267-2800
Email: paulsr@dnr.state.wi.us

- Mr. Terry Carroll and Mr. Tom Baudhuin
Minergy Corporation
1512 S. Commercial Street, P.O. Box 375
Neenah, Wisconsin 54957
Phone: 920/727-1411
Fax: 920/727-1418
Email: tcarroll@minergy.com
Email: tbaudhuin@minergy.com
- Mr. Gary Folk
GE Energy and Environmental Research Corporation
1001 Aviation Parkway, Suite 100
Morrisville, North Carolina 27560
Phone: 919/460-1060
Fax: 919/460-1944
Email: gary.folk@ge.ps.com
- Dr. Joseph Dauchy and Mr. Ken Brown
Tetra Tech EM Inc.
330 S. Executive Drive
Suite 203
Brookfield, Wisconsin 53005
Phone: 262/821-5894
Fax: 262/821-5946
Email: dauchyj@ttemi.com
Email: brownk@ttemi.com
- Dr. Kenneth Partymiller
Tetra Tech EM Inc.
5326 Paris Pike
Georgetown, Kentucky 40324
Phone: 502/867-1397
Fax: 502/867-0423
Email: partykm@aol.com
- Dr. Greg Swanson
Tetra Tech EM, Inc.
591 Camino de la Reina
San Diego, California 92108
Phone: 619/718-9676
Fax: 619/718-9698
Email: swansog@ttemi.com

Section: 2.0
Revision: 2
Date: 7/24/01
Page: 2-9

- Mr. Jim Hahnenberg and Mr. Scott Cieniawski
U.S. Environmental Protection Agency Region 5
77 West Jackson Boulevard
Chicago, Illinois 60604-3507

Phone: 312/353-9184 and 312/886-4040
Email: hahnenberg.james@epa.gov
Email: cieniawski.scott@epa.gov
- Mr. Dennis Johnson
Hazen Research, Inc.
4601 Indiana Street
Golden, Colorado 80403
Phone: 303/279-4501
Fax: 303/278-1528
Email: johnsondm@hazenusa.com
- Mr. Chris Cornwell
Paradigm Analytical Laboratories
2627 Northchase Parkway SE
Wilmington, North Carolina 28405
Phone: 910/350-2839
Fax: 910/350-8911
Email: ccornwell@paradigmanalytical.com
- Mr. Steve Hall
Kiber/Kemron Environmental Services
3145 Medlock Bridge Road
Norcross, Georgia 30071
Phone: 770/242-4090 ext.239
Fax: 770/242-9198
Email: stevehall@kiber.com

3.0 EVALUATION DESIGN

The evaluation design provides details of EPA's proposed plans to accomplish the overall project objectives, which include determining the effectiveness and efficiency of Minergy's GFT in reducing PCB concentrations in contaminated river sediment and providing a product that qualifies for beneficial reuse. The following sections describe the operating parameters, sampling strategy, methods of data analysis, and presentation of results.

3.1 OPERATING PARAMETERS AND SAMPLING STRATEGY

The operating parameters and sampling strategy for the Minergy's GFT demonstration are discussed in the following sections.

3.1.1 GFT System Installation and Operating Conditions

The GFT system will be installed, maintained, and operated by Minergy. The GFT system consists of two-part process: drying to reduce the moisture content to 10 percent or less, then melting the sediment particles to form glass. Minergy claims the process destroys PCB contaminants. WDNR collected and analyzed river sediment when dredging operations were conducted at SMU 56/57 on the Fox River. This sediment will be used as the feedstock for the demonstration.

As a precursor to the full-scale demonstration and to evaluate the effectiveness of the drying operation, a pilot-scale dryer will be tested using a sample portion of the sediment stored in the roll-off containers at the Brown County Landfill. Four 55-gallon drums have been filled with a composited mixture of sediment from the four roll-off containers. This sample mixture will be sent to Hazen in Golden, Colorado, to be used as feedstock in the pilot-scale dryer. The dryer is used to reduce the moisture content of the sediment from 50 percent to 10 percent or less.

The remainder of the river sediment (about 60 cubic yards) will be dried in a dryer located at the Minergy facility. The melter unit, currently being designed and constructed, will be located at the Minergy facility in Winneconne, Wisconsin.

Minergy will establish operating conditions for the melter system based on professional judgment and experience. The technology demonstration is expected to last two weeks in Spring 2001.

3.1.2 Sediment and Product Sampling To Meet Primary Objectives

PCBs are a family of 209 specific compounds, or congeners, which were manufactured by the chemical addition of chlorine to biphenyl. For this technology evaluation program, the fate of specific congeners will be determined. Table 1.1 contains the target list of congeners for this project. These congeners were selected as the most prevalent PCBs present in environmental samples collected from the Fox River area.

It is imperative that the term, PCB, used in this document is not confused with the definition referring to commercial mixtures which were manufactured by chlorinating biphenyl to specific weight percentages. Minor variations in the manufacturing process caused batch-to-batch differences in the exact proportions of each congener present in these technical mixtures. These mixtures are commonly referred to as the trade name Aroclor®. Samples collected to evaluate the primary objectives will be analyzed for specific congeners, not Aroclor® mixtures.

The primary objectives for this technology evaluation are as follows:

- To determine the treatment efficiency (TE) of PCBs in the Minergy GFT in dredged-and-dewatered river sediment

- To determine whether the GFT aggregate product meets the criteria for beneficial reuse under relevant federal and state regulations. The aggregate product will be judged to be beneficial with respect to each metal or PCB if the 95 percent upper confidence bound (UCB) for the estimated mean (of each metal or PCB) is less than the federal or state regulatory requirements, as applicable.

To address the primary objectives of this evaluation, pre- and post-treatment samples will be collected to (1) characterize the PCB content of the dried sediment before processing in the melter and (2) characterize the PCB content in a sample of melter aggregate product and ground aggregate product. The environmental fate of the PCBs contained in the dried sediment will also be evaluated by sampling for PCB concentrations in the melter process air exhaust and wastewater (condensate) streams. Table 3-1 summarizes the dryer and melter test sampling protocols (see Figures 4-1 and 4-2 for sampling locations).

3.1.2.1 Pilot-Test Sampling

Sediment will be mixed before being placed in the dryer. Sediment will first be blended and split into smaller sample sizes (5- to 7-gallon buckets). Blending will be accomplished by coning, quartering, and repiling the sediment three times using shovels on an impervious, bermed pad. The material will then be mixed thoroughly using a pug mill. After the material has been blended, it will be spread out in a circle on the pad, and 5- to 7-gallon buckets will be filled by taking shovel samples from various points around the edge of the circle. This method of sampling will continue until all material has been placed into the buckets.

All of the buckets will be weighed on a scale before drying to determine mass of sediment processed through the dryer. After processing a portion of the sediment in buckets through the dryer, the unprocessed buckets, as well as the empty buckets will be weighed again to obtain a measurement of the amount of undried sediment. The amount of undried sediment will be subtracted from the total amount of sediment sorted into buckets to obtain the amount of sediment processed through the dryer. The scale

Section: 3.0
Revision: 2
Date: 7/24/01
Page: 3-4

TABLE 3-1
DEMONSTRATION SAMPLES TO BE COLLECTED

Samples and Locations ^a	Sampling Type	Analytical Parameter	Number of Dryer Samples		Number of Melter Samples		Total Number of Samples
			Objectives	QA/QC	Objectives	QA/QC	
Sediment and Glass (S1, S2, A, B, C, D)	Composite	PCBs, PCDDs and PCDFs	20	7	44	24	95
		VOCs	NA	NA	4	3	7
		SVOCs	12	4	12	9	37
		Metals	14	4	18	9	45
Water (W1, E, F, G)	Composite	PCBs, PCDDs and PCDFs	10	3	10	3	26
		SVOCs	6	3	8	3	20
		Metals	4	1	8	3	16
Air (G1, H, I, J)	Grab	PCBs, PCDDs and PCDFs	6	NA	18	4	28
		SVOCs	6	NA	7	2	15
		Metals	4	NA	7	6	17
		VOCs	NA	NA	12	4	16
		HCl/Cl ₂	NA	NA	4	5	9
Flux (K)	Grab	PCBs, PCDDs and PCDFs	NA	NA	4	NA	4
		SVOCs	NA	NA	2	NA	2
		Metals	NA	NA	2	NA	2

Notes: NA Not applicable

QA/QC quality assurance/quality control

^a see Figures 4-1 and 4-2 for sample locations (A, B, C, D, E, F, G1, H, I, J, K, W1, S1, and S2)

used to weigh the sediment buckets will be calibrated and certified by weighing an object of known weight before weighing the sediment buckets. The object of known weight will be weighed again after the sediment buckets are weighed to certify that the scale was accurate during the test.

After the sediment has been mixed, the individual 5- to 7-gallon buckets will be used to transfer the material to the dryer continuously throughout each day (8 hours) of the pilot test. After a period of warmup each day, the sediment will be processed continuously through the dryer. Drying sediment for a period of hours will equal 1 "batch." A 15-minute sample setup period will occur between each 2-hour batch. During the setup, sediment will continue to be dried, samples will be collected and containerized, and preparations will be made for the next batch. The through-put for the dryer is about 5 to 15 pounds per hour, for three batches per day; six batches will be processed in two days.

The sampling program for the pilot-test sediment will consist of four samples collected from each of the six batches after the dryer. The four grab samples will be composited in a stainless-steel bowl to form one sample for each batch. About 16 ounces of sediment from each composite sample will be collected and analyzed for PCBs, dioxins/furans, metals including mercury, and SVOCs, both before and after drying in Hazen's pilot-scale dryer. The sample data will therefore consist of 12 pre- and post-dryer treated composite samples, collected across six experimental runs.

Flue gas from the pilot-scale dryer will be sampled. Oxygen, carbon monoxide, and carbon dioxide in the exhaust from the drying process will be measured using EPA Method 3 (ORSAT analyzer). The samples collected during each run will be analyzed to determine what, if any, PCBs, dioxins/furans, SVOCs, or metals are lost during the drying process. Volatilization of compounds with high vapor pressures or boiling points below the drying temperature are the only species expected to be collected. Therefore, the low concentration levels of many organic compounds will require extended run times to collect measurable quantities of the selected pollutants. The high moisture content of the process stream will be reduced in the condensing system and should not effect the collection of the flue gas samples. The condensate will be collected and analyzed as a separate process stream.

The non-condensable flue gas will be sampled for metals including mercury, SVOCs, and for dioxins/furans and PCB congeners. Samples for hydrogen chloride, and chlorine will not be collected. Sampling of these analytes would not be possible because the entire available volume of flue gas from the bench-scale unit will be sampled during the collection of each sampling train. The duct for the flue gas will be less than 4 inches in diameter. Therefore, given the low volume of flue gas and the small diameter of the duct, a sample of flue gas will be collected between sampling runs using a Tedlar bag. These bagged flue gas samples will be used to determine carbon monoxide, carbon dioxide, and oxygen using an ORSAT analyzer; ambient conditions are anticipated in the samples.

Any condensate generated during the drying process will be collected and sampled for PCBs, dioxins/furans, SVOCs, and metals. A 2-hour batch of sediment should generate about 1 gallon of liquid. The liquid will be collected in a condensate trap, as a part of the flue gas exhaust system. Grab samples will be collected from the trap during the batch run and composited. A total of six condensate samples will be collected and analyzed for metals, SVOCs, PCBs, and dioxins/furans.

3.1.2.2 Melter Treatment Sampling

After the Hazen dryer pilot test is complete and data have been analyzed, the sediment stored at the Brown County Landfill will be shipped to the Minergy facility in Winneconne, Wisconsin. The sediment will be dried in a commercial-scale dryer before being used as feedstock for the melter system. The sediment will have previously been mixed with a backhoe and will be further mixed during the loading procedure that transfers the sediment from the roll-off boxes to 55-gallon barrels. The loading procedure will incorporate sediment from representatively distributed locations within the roll-off boxes and combine it in 55-gallon barrels. This approach will be done to further mix the sediment and ensure that blocks of unmixed sediment are not processed.

After drying, the sediment will be crushed to reduce clumps, then magnetically screened, to remove iron, and placed in a mixer for flux addition. Sediment will be fed into the melter continuously in 187-pound

batches of sediment and flux. Composite samples of the sediment will be collected before and after it is mixed with flux material. Grab samples of the flux will confirm the absence of contaminants in the material. Six corresponding time-sequenced post-melter composite samples of the aggregate from the same six distinct batches will be collected after discharge from the melter. This will correspond with the amount of material expected to pass through the melter over a 6-hour period. One composite sample will be collected over each day of the six-day evaluation. The sampling sequence will be based on the expected residence time of the melting process. Hence, the sample data will consist of six pre- and post-melter, paired composite samples. The duration of the melter test will correspond to the amount of sediment and the time necessary to process it through the melter.

Each composite sample will be analyzed for concentrations of PCBs, dioxins/furans, metals including mercury, and SVOCs. VOC concentrations will be analyzed in the dried sediment (pre-melter) samples and the flue gas (post-melter) samples to detect the formation of any VOC-breakdown products resulting from the melting process. Aggregate samples will be subjected to a suitable extraction before analysis of COC concentrations. A stainless-steel scoop will be used to extract about 24 ounces of sediment for each of the composite samples. The sediment samples will be deposited directly into a stainless-steel bowl where they will be mechanically blended to create one dried sediment mixture. From this mixture, three 4-ounce samples will be extracted and placed in individually cleaned, soil sample jars. Glass samples for analyses will also be collected in separate 8-ounce soil sample jars. The remaining material in the sample mixture bowl will be placed in soil sample jars to serve as a backup if necessary.

Following extraction, post-treatment aggregate will be analyzed for COC concentrations to determine whether the aggregate will qualify for beneficial reuse. Composite samples of the aggregate will be duplicated so that samples from each of the six runs can be analyzed by the prescribed methods, and the duplicate composite samples can be crushed and subjected to suitable extraction. One-half of the glass product sample will be subject to analysis of PCBs, dioxins/furans, metals including mercury, and SVOCs using a suitable extraction method and EPA methods for sample preparation, cleanup, and quantification. The remaining half of the glass product sample will be ground to a #200-mesh screen diameter and

analyzed for PCBs, dioxins/furans, metals including mercury, and SVOCs using a suitable extraction method and EPA methods for sample preparation, cleanup, and quantification. A total of 12 composite samples of the post-treatment aggregate will be collected.

To determine potential PCB losses to the environment as a result of Minergy's melter process, the melter exhaust gas will be sampled and analyzed. In addition, quench water will be analyzed for any residual PCBs. These sampling procedures will provide the analytical basis for quantifying the first primary objective: the treatment efficiency of PCBs from the dredged-and-dewatered river sediment using the Minergy GFT process. The second primary objective – determining whether the final aggregate product meets the criteria for beneficial reuse based on PCB and metal concentrations being less than state and federal regulatory requirements – is also evaluated in the preceding sampling approach. Additional sampling required to evaluate certain secondary objectives is discussed below.

3.1.3 Sediment and Product Sampling To Meet Secondary Objectives

The secondary objectives for this technology evaluation are as follows:

- Determine the unit cost of operating the GFT on dredged-and-dewatered river sediment
- Quantify the organic and inorganic contaminant losses resulting from the existing or alternative drying process used to dry the dredged-and-dewatered river sediment
- Characterize organic and inorganic constituents in all GFT process input and output streams

The attainment of the first secondary objective does not rely on a sampling protocol but rather is achieved through an accounting function. To determine the unit cost of removing the PCBs and the organic and inorganic contaminants from the river sediment, the capital and the fixed and variable operating costs associated with both the pilot-scale dryer and the melter will be collected from Hazen and Minergy. Minergy will work with EPA to forecast the equivalent costs for production-sized dryers and melters.

Attendant air pollution control devices or wastewater treatment systems required for the units to maintain full compliance with federal and state emissions and effluent discharge limitations will be included in the capital and variable cost estimates. Specifically, this objective will be achieved by assessing the following expense categories:

- Mobilization
- Site preparation
- Permitting and regulatory compliance
- Capital equipment/equipment lease
- Startup
- Labor
- Consumable material
- Utilities
- GFT system implementability
- Waste disposal
- Residual waste shipping and handling
- Analytical services
- Demobilization

The above parameters will be measured and calculated for the pilot-scale drying test. The costs for utilities, labor, waste disposal, preparation, and other tasks will be examined by Minergy and EPA to estimate the cost per ton for the full-scale treatment of sediment.

The second secondary objective will be evaluated based on the data collected during the pilot-scale drying test conducted at the Hazen facility. The battery of samples collected and detailed in the primary objective sampling will permit the quantification of organic and inorganic content before and after the drying process. The concentrations of PCBs, dioxins/furans, metals including mercury, and SVOCs will be used to determine any contaminant losses due to the drying process. The analysis and quantification will be performed using the data collected during the pilot-scale dryer test at the Hazen facility, and the results will be extrapolated to estimate the potential losses during commercial dryer operations.

The third secondary objective will combine data from all the input and output streams of the GFT process and will characterize the results to assess the efficiency and competence of the GFT process.

3.2 DATA ANALYSIS AND PRESENTATION OF RESULTS

The data analysis, like the sampling plan, is designed to address the objectives of the project. Descriptive statistics, statistical tests, or construction of confidence intervals will be used to evaluate the populations from which the samples were collected. Descriptive statistics will include calculation of the range, upper and lower values, mean, median, and variance. The statistical test proposed for this investigation is the paired t-test for systematic random samples. Confidence intervals will be based on the student's t distribution. An analysis of variance test will be performed on the sediment sample results to characterize the variability of the sample population.

As noted in EPA guidance (EPA 1998), "All statistical tests make assumptions about the data."

Typically, such assumptions include those regarding population distribution, equality of variance, the percentage of censored (or nondetect) data, independence of the data, and the presence or absence of outliers. Therefore, the validity of the assumptions and the sensitivity of a particular test to violations of the assumptions should be evaluated upon initial review of the data.

Accordingly, all data will be reviewed before subjecting it to statistical tests or other methods. If necessary, other statistical tests or analytical methods better suited to the data may be substituted for the tests and methods discussed in the following paragraphs. Any tests used will satisfy project objectives.

P1 To determine the treatment efficiency of PCBs of the Minergy GFT in dredged-and-dewatered river sediment

The first (pilot-scale dryer) and second (melter) phases of the demonstration will generate data needed to evaluate the TE of the process. During the first phase, six composite samples will be collected from the pre-dryer sediment (one from each day) and six composite samples will be collected from the post-dryer sediment; the 12 samples will be analyzed for PCBs.

During the second phase, similar data will be collected from the pre- and post-melter material. The dried sediment will become feedstock for the melter system. The duration of the melter test will correspond to the amount of sediment and the time necessary to process it through the melter. For the purpose of sampling the pre-melted sediment and glass aggregate, a composite sample of the sediment will be collected for each day of the melter demonstration. A grab sample from each 187-pound batch (170 pounds of dried sediment and 17 pounds of flux) after it is mixed with flux material will be composited to make the sample. This will correspond with the amount of material expected to pass through the melter over a 6-hour period. Six corresponding time-sequenced post-melter composite samples of the aggregate from the same six distinct runs will also be collected after discharge from the melter. The sample data will consist of six pre- and post-melter, paired, composite samples.

The TE of PCBs for each phase of the Minergy GFT, and the overall TE will be estimated using the sample data, along with 95 percent confidence intervals. Each TE will be estimated using the following formula:

$$TE = 100\% \times (W_{in} - W_{out}) / W_{in}$$

where W_{in} = geometric mean PCB input level:

For the overall GFT process (drying and melting), W_{in} represents the geometric mean of the PCB concentration of the dewatered sediment; for the melting system only W_{in} represents the geometric mean of the PCB concentration of the dried sediment

W_{out} = geometric mean PCB output level:

For the drying system only, W_{out} represents the geometric mean for the PCB concentration of the dried sediment; for the overall GFT system, W_{out} represents the geometric mean for the combined PCB concentrations of the process flue gas stream, the quench water stream, and the aggregate product.

The estimated difference between the post- and pre-treatment log means () will then be used to estimate the geometric mean TE (TE_{GM}) using the following formula:

$$TE_{GM} = 100\% \times (1 - \exp())$$

which is mathematically equivalent to the TE formula stated previously.

The calculated standard error of the estimated difference between the post- and pre-treatment log means will then be used to construct a 95 percent confidence interval, and this confidence interval will be back-transformed to produce an equivalent 95 percent confidence interval for the TE_{GM} estimate.

The total TE for the entire process will be estimated by pooling the two individual estimated differences between the post- and pre-treatment log means, using the following formula:

$$TE_{GM,Total} = 100\% \times (1 - \exp(-\tilde{a}))$$

An *approximate* 95 percent confidence interval will also be calculated for this estimate by pooling the two individual standard error estimates and appropriately back-transforming the resulting confidence limits (associated with the \tilde{a} estimate).

P2 To determine whether the GFT aggregate product meets the criteria for beneficial reuse under relevant federal and state regulations. The aggregate product will be judged to be beneficial with respect to each metal or PCB if the 95 percent UCB for the estimated mean (of each metal or PCB) is less than the federal or state regulatory requirements, as applicable.

Suitable extraction tests will be conducted on the aggregate product in both a coarse and ground state. Results of the extraction tests will be evaluated against state and federal criteria levels for defining a hazardous waste. The aggregate product will be judged to be beneficial with respect to each metal or total PCBs if the 95 percent UCB for the estimated mean (of each metal or total PCBs) is found to be below the federal or state regulatory requirements, as applicable. The WDNR, for example, will compare the results of these tests against their criteria for beneficial reuse and PCB concentration as listed in Table 1-2.

Test Statistic 1: Approximate Normality Assumed

The 95 percent UCB associated with the mean metal or PCB level for the final coarse glass aggregate data will be estimated using the calculated sample mean and variance defined as follows:

$$UCB = \tilde{a} + t_{0.05, n-1} (s/n^{0.5})$$

$$\tilde{a} = 3(\bar{x} / n)$$

$$s^2 = 3[(\bar{y}_i - \bar{a})^2 / (n-1)]$$

$$\bar{y}_i = y_{i,\text{post}}$$

$t_{0.05, n-1}$ = 0.05 level of a t -distribution with $n-1$ degrees of freedom

for $i = 1, 2, \dots, 6$ ($n = 6$ sample replications) and

y = post treatment aggregate PCB and metal sample data

The resulting UCB will be used to determine if the resultant aggregate PCB and metal sample data meet federal or state regulations for beneficial reuse. If the UCB is less than the federal or state guidelines, then the aggregate will be judged to be beneficial.

Test Statistic 2: Non-Normally Distributed Data

The 95 percent UCB associated with the mean metal or PCB level for the final glass aggregate data will be estimated using a bootstrap resampling approach: 5,000 samples of size 6 will be drawn with replacement from the post-treated sediment PCB and metal data, so that 5,000 distinct estimates of the mean levels can be determined. This bootstrap distribution of mean estimates will then be used to determine (1) the overall bootstrap sample mean and (2) the upper 95 percent value of the bootstrap distribution, which represents the 95 percent UCB for the overall sample mean. The overall sample mean will be determined by computing an arithmetic average of the 5,000 individual mean estimates. The upper 95% value of the bootstrap distribution will be determined by ordering the 5,000 estimates (from low to high) and determining the value of the 95th quantile.

The resulting UCB will be used to determine if the aggregate PCB and metal sample data meet federal or state regulations for beneficial reuse. If the UCB is less than the federal or state guidelines, then the aggregate will be judged to be beneficial.

Coarse versus crushed glass aggregate differences

The difference between the mean metal or PCB levels for the final coarse versus crushed sediment PCB and metal sample data will be estimated using the calculated sample mean and variance from a paired t-test statistic. The resulting t-test statistic will be used to determine if a statistically significant difference exists between the mean coarse and crushed aggregate sample levels at the 0.05 significance level. Since the crushing of the aggregate may release higher amounts of harmful metals or PCBs, a one-sided t-test will be used. Therefore, the null and alternative hypotheses will be defined as follows:

$$\begin{aligned}H_o: & \quad \tilde{a} \# 0 \\H_a: & \quad \tilde{a} > 0\end{aligned}$$

Rejection of the null hypothesis will imply that the post-treated mean PCB or metal levels associated with the crushed sample aggregate exceed the same post-treated mean PCB or metal levels associated with the coarse sample aggregate.

S1 Determine the unit cost of operating the GFT on dredged-and-dewatered river sediment.

The unit cost of removing the PCBs and the organic and inorganic contaminants from the river sediment, capital, fixed, and variable operating costs associated with both the pilot-scale dryer and the melter will be determined by Hazen and Minergy. The pilot-scale estimates will be used to forecast the equivalent costs for production-sized dryers and melters. This secondary objective will be achieved by assessing several expense categories.

S2 Quantify the organic and inorganic contaminant losses resulting from the existing or alternative drying process used to dry the dredged-and-dewatered river sediment.

The battery of samples collected and detailed above will permit the quantification of organic and inorganic content before and after the drying process. The concentrations of PCBs, dioxins/furans, metals including mercury, and SVOCs will be used to determine any contaminant losses due to the drying process. The analysis and quantification will be performed using the data collected during pilot-scale dryer test at the Hazen facility, and the results will be extrapolated to estimate potential losses during the commercial dryer operations.

The results of the pilot-scale dryer test will be evaluated in the same manner as that used to obtain primary objective 1. Rather than describe the details of the statistical evaluation again, the 95 percent confidence intervals of the pre- and post-dryer samples will be constructed using data assumptions as described above. The results of all the pre- and post-dryer samples will be compared to assess whether any contaminant losses have occurred.

S3 Characterize organic and inorganic constituents in all GFT process input and output streams

The third secondary objective will combine data from all the input and output streams of the GFT process and characterize the results to assess the GFT process. In addition, VOCs analysis will be conducted on both pre- and post- melter samples to evaluate the potential production of volatile compounds in the melting process.

As in the second secondary objective, the results of all the samples collected during both the pilot-scale dryer test and the melter test will be evaluated in a similar manner as that used to obtain primary objective 1. The 95 percent confidence intervals will be calculated with the same formula described above. The paired results of the statistical evaluation (pre- versus post-treated samples) will be compared to characterize any losses or changes.

4.0 FIELD SAMPLING METHODOLOGIES AND PROCEDURES

The main objective of the planned sampling and monitoring program is to provide sufficient data to allow EPA to evaluate the performance of the Minergy GFT process and to meet the primary and secondary demonstration objectives discussed in Section 1.4. The goal of the planned sampling program is to collect and analyze samples of sufficient number and quality such that the results accurately reflect the performance of the treatment system during the demonstration.

Several sampling objectives must be met for this demonstration to produce well documented, defensible data that are of known and reproducible quality. General sampling objectives for the demonstration of the technology are as follows:

- Collect representative samples
- Preserve and ship samples in a manner designed to ensure sample integrity and continued representativeness
- Maintain proper chain-of-custody control of all samples, from collection to analysis
- Follow QA/QC procedures appropriate for EPA Office of Research and Development (ORD) Applied Research projects

The following sections discuss the site locations, on-site facilities to be used during sample collection, and sediment and flue gas sampling procedures.

4.1 SITE LOCATION

The project will be conducted in two phases in two separate site locations. The first phase consists of drying the sediment in Golden, Colorado. The second phase consists of melting the dried sediment in Winneconne, Wisconsin.

A pilot-scale Holoflite® Processor will be used to determine the operating parameters for a commercial unit to dry the bulk of the sediment. The pilot-scale unit is located at the Hazen facility outside of Golden, Colorado. Because both the pilot-scale unit and any potential commercial unit operate under the same principle, the air sampling program will be conducted for the pilot-scale dryer only.

The pilot-scale melter unit will be located at the Minergy facility in Winneconne, Wisconsin. The melter is currently being designed and constructed by Minergy. After being transported to Minergy's facility, the sediment will be dried in a drum dryer, then processed through the melter.

4.2 ON-SITE FACILITIES FOR THE FIELD SAMPLING TEAM

On-site facilities will be available for use by the field sampling team for both phases of the demonstration. Testing facilities available for the drying process and GFT phases are discussed below.

4.2.1 DRYING PROCESS TESTING FACILITIES

On-site facilities required for field sampling during the drying phase include (1) laboratory space for sample train assembly, preparation, recovery, as well as reduction of field data; (2) a sample storage refrigerator or coolers; (3) electrical connections sufficient to provide power to the sample metering consoles; and (4) sanitary facilities, including running water.

The laboratory facilities at the site will be made available to the field sampling team. Therefore, a temporary mobile laboratory will not be required. Condensate samples and sediment samples will be collected and stored in a refrigerator or packed in an ice cooler. Air samples will be stored according to the procedures specified in each method, until they are shipped to the off-site laboratory. Electrical utilities will be required for sample collection, and electrical hookups will be required for sample recovery and storage. Due to the short duration of the tests and the limited number of personnel, the sanitary facilities already on-site will be adequate for use by the sampling team.

Communication with the on-site team during the sampling program will be made by telephone. If an on-site telephone is not available, then a cellular telephone will be supplied by GE EER.

4.2.2 GLASS FURNACE TECHNOLOGY PROCESS TESTING FACILITIES

On-site facilities required for field sampling during the melter phase include (1) a mobile laboratory for sample train assembly, preparation, and recovery, as well as data reduction; (2) a mobile CEMS laboratory for collecting and analyzing process gas samples; (3) a sample storage refrigerator or coolers; (4) electrical connections sufficient to provide power to the Continuous Emission Monitor (CEM) truck and the sample metering consoles; and (5) sanitary facilities, including running water.

No laboratory facilities are available at the site. Therefore, a temporary mobile laboratory will be required to assemble sampling trains and perform sample recoveries and other activities. Water samples and sediment samples will be collected and stored in a refrigerator or packed in an ice cooler. Air samples will be stored according to procedures specified in each method until they are shipped to the off-site laboratory. Electrical utilities will be required at the sampling locations, and electrical hookups will be needed for sample recovery and storage. Due to the short duration of these tests and the limited number of personnel, the sanitary facilities already on site will be adequate for use by the sampling team.

The on-site team will communicate by telephone during the sampling event. If a telephone is not available in the on-site trailer that can be used for off-site contact, a cellular telephone will be supplied by GE EER.

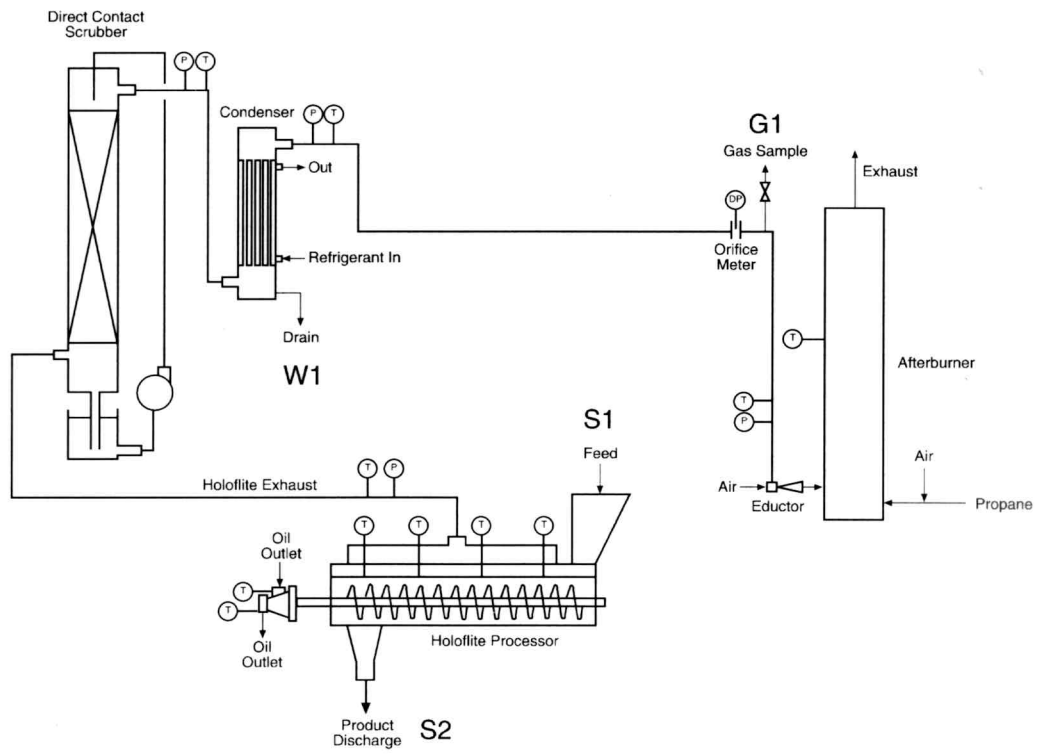
Communication among the GE EER project manager, process personnel, and sampling personnel will be accomplished by wireless two-way radios, unless all personnel are within close enough proximity to communicate effectively. Each team leader and the field coordinator will be equipped with a radio. This will ensure that the personnel assigned to each station can be in immediate contact with the field coordinator and with each other if problems arise during the testing program.

4.3 SAMPLING LOCATIONS

Figure 4-1 provides the general schematics and sampling locations of the drying process that will be employed during the first phase of the project. Figure 4-2 provides the general schematics and sampling locations of the glass melter that will be used during the second phase of the project. The sampling locations, matrices, and identifications of locations shown in Figure 4-1 are described in Table 4-1. The sampling locations, matrices, and identifications of locations shown in Figure 4-2 are described in Table 4-2.

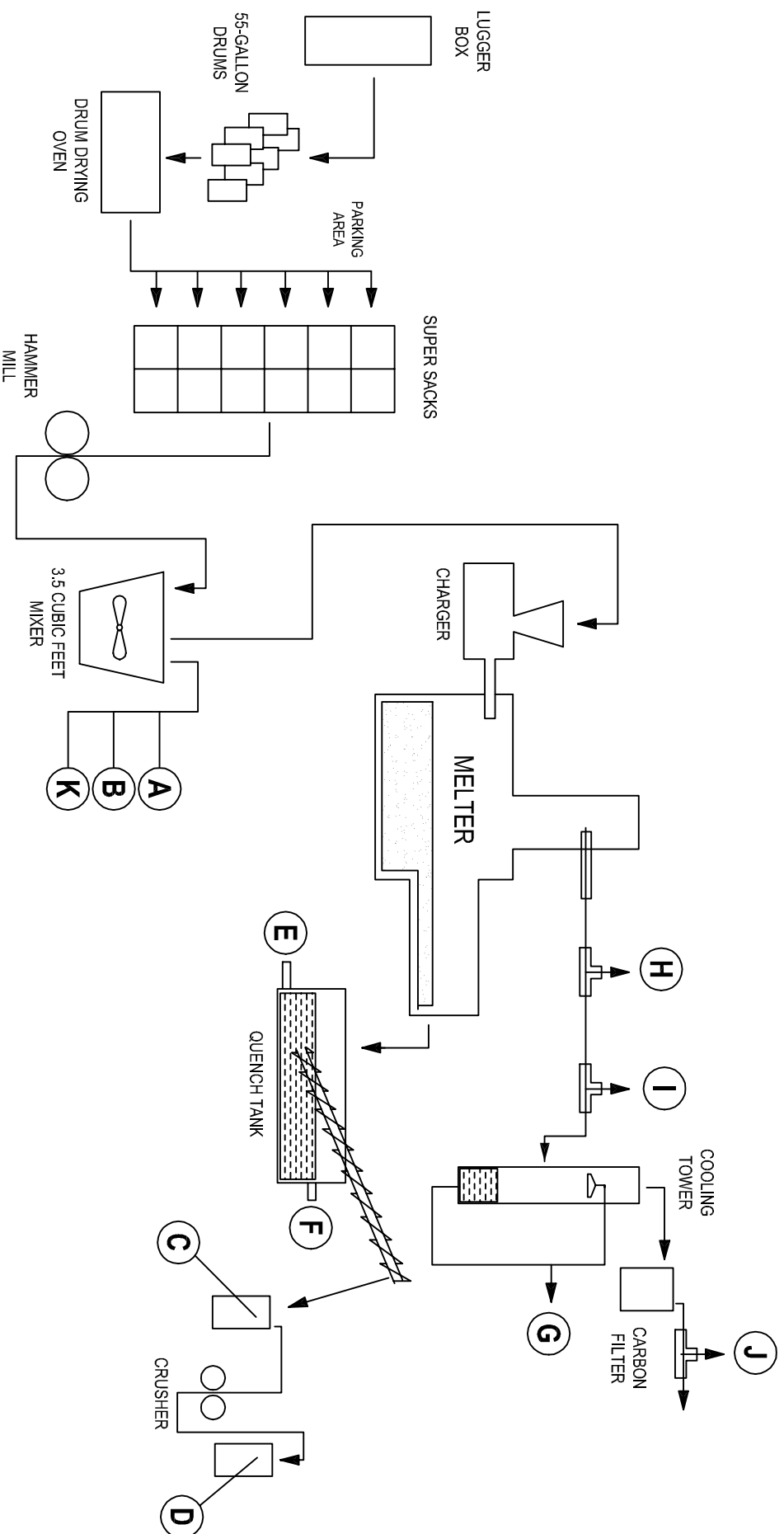
Table 4-1 Sampling Locations and Identifications During Drying Process

Location ID	Description of Sampling Location	Sample Matrix
G1	Flue gas from drying process	Gaseous
S1	Composite of sediment prior to drying	Solid
S2	Composite of sediment after drying	Solid
W1	Water (condensate) removed during the drying process	Aqueous



Schematic diagram of the pilot scale dryer.

FIGURE 4-1. SAMPLING LOCATIONS FOR THE DRYING PROCESS



US EPA SITE PROGRAM
MINERGY DEMONSTRATION

FIGURE #4-2
SCHEMATIC OF GFT PROCESS



TETRA TECH EM INC.

TABLE 4-2

KEY TO SAMPLING LOCATIONS FOR MINERGY MELTING OPERATION

SAMPLING LOCATION	MATRIX	SAMPLE TYPE	NUMBER OF SAMPLES PER COMPOSITE	ANALYSES					DESCRIPTION
				PARAMETER	NUMBER OF SAMPLES	NUMBER OF QUALITY CONTROL SAMPLES			
						Dup/ FB	MS/ MSD	Reagent Blank	
A	Soil	Composite	28	PCB	6	1	2	-	Dried, mixed sediment without flux addition To determine the variability of the material Collected from sacks
B	Soil	Composite	24	PCB	6	1	2	-	Dried, mixed sediment with flux addition To determine the chemical characteristics of the dried sediments prior to the melter Collected over 6-hour periods
	Soil	Composite	24	Dioxin/Furan	4	1	2	-	
	Soil	Composite	24	SVOC	4	1	2	-	
	Soil	Composite	24	Metals	4	1	2	-	
	Soil	Composite	24	Mercury	6	-	-	-	
	Soil	Composite	24	VOC	4	1	2	-	
C	Glass	Composite	24	PCB	6	1	2	-	Glass material from the melter To determine the chemical characteristics of the leachate extracted off the glass surface Collected over 6-hour period
	Glass	Composite	24	Dioxin/Furan	4	1	2	-	
	Glass	Composite	24	SVOC	4	1	2	-	
	Glass	Composite	24	Metals	4	1	2	-	
	Glass	Composite	24	Mercury	6	-	-	-	

TABLE 4-2 (continued)

KEY TO SAMPLING LOCATIONS FOR MINERGY MELTING OPERATION

SAMPLE LOCATION	MATRIX	SAMPLE TYPE	NUMBER OF SAMPLES PER COMPOSITE	ANALYSES					DESCRIPTION
				PARAMETER	NUMBER OF SAMPLES	NUMBER OF QUALITY CONTROL SAMPLES			
						Dup/ FB	MS/ MSD	Reagent Blank	
D	Glass (crushed)	Composite	24	PCB	12	2	4	-	Glass material from the melter Crushed to <200 mesh To determine the chemical characteristics of the leachate extracted off the glass surface Collected over 6-hour period
	Glass (crushed)	Composite	24	Dioxin/Furan	6	1	2	-	
	Glass (crushed)	Composite	24	SVOC	4	1	2	-	
	Glass (crushed)	Composite	24	Metals	4	1	2	-	
	Glass (crushed)	Composite	24	Mercury	6	-	-	-	
E	Water	Grab	NA	PCB	2	-	-	-	City Water To determine the quality of the water entering the quench tank Collected at the beginning and the end of the 6-day period
	Water	Grab	NA	SVOC	2	-	-	-	
	Water	Grab	NA	Metals	2	-	-	-	

TABLE 4-2 (continued)

KEY TO SAMPLING LOCATIONS FOR MINERGY MELTING OPERATION

SAMPLE LOCATION	MATRIX	SAMPLE TYPE	NUMBER OF SAMPLES PER COMPOSITE	ANALYSES					DESCRIPTION
				PARAMETER	NUMBER OF SAMPLES	NUMBER OF QUALITY CONTROL SAMPLES			
						Dup/ FB	MS/ MSD	Reagent Blank	
F	Water	Composite	12	PCB	6	1	2	-	Quench Water To determine the quality of the water exiting the quench tank Collected over 6-hour periods
	Water	Composite	12	SVOC	4	1	2	-	
	Water	Composite	12	Metals	4	1	2	-	
G	Water	Grab	NA	PCB	2	-	-	-	Discharge from Cooling Tower To determine the quality of the water discharged Collected at the beginning and end of the 6-day period
	Water	Grab	NA	SVOC	2	-	-	-	
	Water	Grab	NA	Metals	2	-	-	-	
H	Gas	Grab	NA	PCB	6	1	-	1	Gas Sample Train 1 To determine the chemical characteristics of the materials discharged to the pollution control equipment Collected over 4 hours
	Gas	Grab	NA	Dioxin/Furan	6	1	-	1	

TABLE 4-2 (continued)

KEY TO SAMPLING LOCATIONS FOR MINERGY MELTING OPERATION

SAMPLE LOCATION	MATRIX	SAMPLE TYPE	NUMBER OF SAMPLES PER COMPOSITE	ANALYSES					DESCRIPTION
				PARAMETER	NUMBER OF SAMPLES	NUMBER OF QUALITY CONTROL SAMPLES			
						Dup/ FB	MS/ MSD	Reagent Blank	
I	Gas	Grab	NA	SVOC	4	1	-	1	Gas Sample Train 2 To determine the chemical characteristics of the materials discharged to the pollution control equipment Collected over 4 hours
	Gas	Grab	NA	Metals	4	3	-	3	
	Gas	Grab	NA	HCl/Cl ₂	4	2	-	3	
	Gas	Grab	NA	VOC	12	2	-	2	Collected over 1 hour
J	Gas	Grab	NA	PCB	3	-	-	-	Gas Sample Train To determine the chemical characteristics of the materials discharged to the pollution control equipment Collected over 4 hours
	Gas	Grab	NA	Dioxin/Furan	3	-	-	-	
	Gas	Grab	NA	SVOC	3	-	-	-	
	Gas	Grab	NA	Metals	3	-	-	-	
K	Flux	Grab	NA	PCB	2	-	-	-	Sample of Flux Additive To validate chemical characteristics of any additives to the process Collected from single lot
	Flux	Grab	NA	Dioxin/Furan	2	-	-	-	
	Flux	Grab	NA	SVOC	2	-	-	-	
	Flux	Grab	NA	Metals	2	-	-	-	

Notes: For sampling locations, see Figure 4-2
 Dup - duplicate
 FB - Field blank
 HCl/Cl₂ - Hydrochloric acid/chlorine
 MS/MSD - matrix spike/matrix spike duplicate
 NA - Not Applicable

VOC - Volatile organic compounds
 PCB - Polychlorinated biphenyls
 SVOC - Semivolatile organic compounds

The fate of the target organic compounds and inorganic constituents must be determined to assess the effectiveness of the process in remediating the contaminated matrix. However, completing a mass balance for all parameters is difficult. Currently, there is no database on the speciation of emissions from glass melters vitrifying contaminated sediment. Therefore, the details surrounding the collection of pre-treated materials and the resulting end-product and by-products must be well defined before beginning the project. As currently defined, the project will collect (1) process gas samples to generate detailed speciation data on the emissions from a pilot-scale glass furnace (melter) vitrifying dried river sediment containing PCBs and heavy metals; (2) samples of the quenched melted sediment (aggregate); and (3) samples from any other waste streams identified or as yet undefined, as appropriate. The air sampling strategy is designed to determine the TE of the technology for PCBs. The testing program will be divided into two distinct phases.

Phase I measurements will be conducted during the sediment drying phase of the project. The sediment sampling program will consist of six “wet” composite samples and six “dry” composite samples (one for each batch of sediment processed) to be analyzed for SVOCs, PCBs dioxins and furans, and metals including mercury. The condensate sampling program will consist of six aqueous samples removed from an in-line condenser to be analyzed for SVOCs, PCBs dioxins and furans, and metals including mercury. The six condensate samples will be time-coordinated with the collection of the sediment samples. The exhaust for the drying process will be sampled for SVOCs, PCBs dioxins and furans, and metals including mercury. Six samples will be collected for SVOCs, PCBs, and dioxins and furans. Four samples will be collected for metals including mercury. Oxygen, carbon monoxide, and carbon dioxide in the exhaust from the drying process will be measured using a CEM, if there is sufficient flue gas volume. Otherwise the exhaust will be collected and measured using alternative methodology. The samples collected during each run will be analyzed to determine what, if any, organic species or volatile metals are lost during the drying process.

Phase II will be conducted during the melting process. Sediment measurement will consist of composite samples collected from six days, or runs, analyzed for SVOCs, PCBs, dioxins and furans, and metals

including mercury on the sediment feedstock and the aggregate product (both coarse and ground).

Twenty-four time-sequenced grab samples of the sediment will be collected before input into the melter.

Twenty-four corresponding, time-sequenced, post-melter samples of the aggregate from the same six distinct runs will also be collected after discharge from the melter, with the sampling sequence based on the expected residence time of the melting process. The post-melter composite samples will be subjected to a suitable extraction before analysis of COC concentrations. The post-melter samples will be split, half of which will be crushed to pass through a 200-mesh sieve (75 microns) before being subjected to a suitable extraction and analysis of COC concentrations. The sediment sample data will consist of a total of 18 pre- and post-melter, paired, composite samples collected across six runs.

Flue gas measurement will be collected from six runs for PCBs. Flue gas will be collected in four runs for SVOCs; dioxins/furans; VOCs (consisting of three unique sample trains using the VOST approach, thus yielding 12 data points); hydrogen chloride and chlorine; and metals including mercury. CEM will be used to monitor carbon monoxide, carbon dioxide, nitrogen oxides, sulfur dioxide, oxygen, and total hydrocarbon concentrations. Samples will be collected using sample trains and the CEM from a slip stream of the melter flue gas. The slip stream will be “conditioned” using a heat exchanger to reduce the flue gas temperature from 2,850 °Fahrenheit (F) to less than 400 °F. Particulate sampling will not be performed because particulate matter should not be generated during the melting and vitrification process. Sampling will be conducted in an isokinetic fashion; however, due to the high moisture content (greater than 60 percent) of the oxy-fuel emissions, true isokinetic sampling may not be achievable. Because the flue gas should not contain any particulate matter, and the water content will be gaseous (no droplets anticipated), isokinetic sampling should not be required to collect a representative sample of the gas stream.

4.4 SAMPLING EVENTS

To determine the fate of the organic and inorganic COCs in the sediment samples, air sampling will be conducted during both phases of the process. The first phase of the process will be performed to dry the

river sediment to a moisture level of less than 10% moisture by weight. The second phase of the process will address the melting of the dried river sediment.

EPA will oversee all sampling procedures and collect sediment samples before and after drying for COC characterization. TtEMI will collect normal process data from the dryer, including, but not limited to, the following: sediment feed rates, dryer operational and exit temperatures, condensate rates, and flow rates during the drying procedure.

During the melting process, all waste streams (solid, liquid, and gaseous) will be sampled. EPA will oversee all sampling procedures and collect sediment samples before and after the melting process for COC characterization. TtEMI will collect normal process data from the melter, including, but not limited to, the following: sediment feed rates; oxygen and natural gas flow rates; melter operational and exit temperatures; and flue gas temperatures, velocity, and volume during the vitrification process.

4.5 SEDIMENT SAMPLING PROCEDURES

Sediment sampling procedures for the pilot-scale dryer and the melter process are discussed below.

4.5.1 PILOT-SCALE DRYER SAMPLING

The sampling program for the pilot-test sediment drying will consist of four samples collected from each of the six batches run through the dryer. The four samples will be combined to form a single composite sample for each batch run. This sample method will be replicated to collect samples of the post-dryer sediment for a total of 12 samples.

Four discrete samples will be mixed in a stainless-steel bowl to form one composite sample for each batch. Approximately 16 ounces of the sediment from each of the composite samples will be collected and analyzed for concentrations of PCBs, dioxins/furans, metals including mercury, and SVOCs, both

before and after drying in Hazen's pilot-scale dryer. The sample data will therefore consist of 16 pre- and post-dryer composite samples, collected across eight experimental runs.

Composite sediment samples for each run will be collected in a clean 4-ounce (oz) sample container for each laboratory analysis. The PCBs (EPA Method 680) and dioxins/furans analysis (EPA Method 8290A), the analysis of metals including mercury (EPA Methods 3050B/6010B/7471B), and analysis of SVOCs (EPA Methods 3540C/8270C) each require a single, unpreserved 4-oz glass container of sediment. All sediment samples will be refrigerated to a temperature of 4°Celsius (C) or packed on ice until received by the laboratory.

Field duplicate samples will be collected as QC samples during the sediment evaluation. Duplicate samples will be used to assess the variability of the COC concentrations in the soil and the repeatability of laboratory analyses. Because samplers and composite mixing bowls will be replaced between each run, equipment blanks will not be collected. In addition, a volume of sediment will be collected (three 4-oz jars of pre-dried sediments and three 4-oz jar of post-dried sediment) to allow for MS/MSD analysis by each laboratory.

4.5.2 GLASS FURNACE TECHNOLOGY DEMONSTRATION SAMPLING

After shipment to the Minergy facility in Winneconne, Wisconsin, and dried, the sediment will become feedstock for the melter. The dried sediment will be continuously processed through the melter. Composite samples will be collected for SVOCs, PCBs, dioxins and furans, and metals including mercury, from the sediment feedstock and the aggregate product (both coarse and ground). Twenty-four sequenced grab samples of the sediment will be collected and composited before input into the melter. Twenty-four corresponding, time-sequenced, post-melter samples of the aggregate from the same six distinct runs will also be collected and composited after discharge from the melter. Both of the sequences will be repeated six times. The sampling sequence will be based on the expected residence time of the melting process. All of the post-melter composite samples will be subjected to a suitable extraction before

analysis of COC concentrations. The post-melter samples will be split, half of which will be crushed to pass through a 200-mesh sieve (75 microns). The aggregate and crushed aggregate will be subjected to suitable extraction and analysis of COC concentrations. The sample data will consist of six pre- and post-melter samples, as well as, six crushed samples.

Each composite sample will be analyzed for concentrations of PCBs, dioxins and furans, metals including mercury, and SVOCs. The post-melter composite samples will be subjected to an extraction procedure before analysis of COC concentrations. A stainless-steel scoop will be used to transfer approximately 24 ounces of the sediment directly into a stainless-steel bowl where the sediment will be blended. Three 4-oz composited samples from this mixture will be transferred to clean soil sample jars. Remaining sediment in the sample mixture bowl will be placed in soil jars to serve as a backup, if necessary. The laboratory analyses of these samples using EPA Method 680 will establish the PCB reduction due to treatment in the Minergy melter process.

The aggregate will be analyzed for COC concentrations after suitable extraction to determine whether the aggregate will qualify for beneficial reuse. Composite samples of the treated aggregate will be duplicated so that samples from each of the four runs can be analyzed by the prescribed methods, and the duplicate composite samples can be crushed and subjected to the suitable extraction. One-half of the extruded glass product sample will be subject to analysis of total PCBs, dioxins and furans, metals including mercury, and SVOCs after suitable preparation and EPA methods for sample preparation, quantification, and cleanup. The remaining half of the extruded glass sample will be sufficiently ground to pass through a 200-mesh screen diameter and will be analyzed for PCBs, dioxins/furans, metals including mercury, and SVOCs after suitable preparation and the relative EPA methods for sample preparation, quantification, and cleanup. Therefore, a total of 12 composite samples of the post-treatment aggregate will be collected. Table 4-2 summarizes the sample locations for the melter phase of the testing program.

4.6 FLUE GAS SAMPLING PROCEDURES

To meet the primary and secondary objectives of this testing program, each sampling method must be performed in accordance with the methodology, and data must comply with the QA/QC requirements outlined in this QAPP. To ensure that the program objectives are achieved, sufficient sample volumes must be collected and laboratory minimum detection limits must be met to calculate the fate of the contaminants of interest. For flue gas measurements, the detection limit of a given method is a function of the analytical detection limit and the total sample volume collected in the sampling train. All sampling trains will be operated for a predetermined time period to allow collection of as much sample as possible, based on the methodology and the limitations presented by the stack conditions.

Several site-specific conditions during the drying phase of the demonstration will require modifications to the standard sampling methods to achieve the project's QA objectives. Volatilization of compounds with high vapor pressures or boiling points below the drying temperature are the only species expected to be collected. Therefore, the low concentration levels of many organic compounds will require extended run times to collect measurable quantities of the selected pollutants. The high moisture content of the process stream should be condensed and should not effect the collection of the flue gas samples. Condensate will be collected and analyzed as a separate process stream. Flue gas will be sampled for metals including mercury, SVOCs, and for PCB congeners only. Because organic constituents in the sediment should not be oxidized or combusted in the dryer, samples will not be collected and analyzed for volatile organics and hydrogen chloride and chlorine. The entire available flue gas volume from the pilot-scale unit may be consumed during the collection of two simultaneous sampling trains. The duct for the gas stream will be less than 4 inches in diameter. Given the low volume of flue gas and the small diameter of the duct, isokinetic sampling cannot be performed. If there is sufficient flue gas volume, a sample will be collected in a Tedlar® bag to determine carbon monoxide, carbon dioxide, and oxygen concentrations using an ORSAT analyzer; however, ambient conditions are anticipated in the samples.

Several site-specific conditions during melter phase of the demonstration will require modifications to the standard sampling methods to achieve the projects's QA objectives. The low levels of many organic compounds will require extended run times to collect measurable quantities of the desired compounds. The temperature of the flue gas exiting the melter is expected to be 2,850 °F. Temperatures greater than 1,000 °F requires the use of specially designed probes that can either withstand the extreme temperature or can cool the internal probe liner. A portion of the flue gas flow will be split from the total flow and be reduced in temperature to less than 400 °F prior to sampling. This heat exchange system will eliminate the need for high temperature equipment for sampling, however, it will not address the high moisture (60 percent) concentration in the flue gas. To address the effect the high moisture content will have on the sampling rate, each sampling train will be modified with an "in-stack orifice" to more accurately control the sampling flow rate. The orifice should allow isokinetic conditions to be maintained in the high moisture environment. In addition to the orifice, a cyclone or demister will be added between the sampling train filter assembly and the adsorbent traps (M23, M0010, and M0031) to reduce the volume of water on the traps. For the M0060 (Metals) and the M0050 (hydrogen chloride and chlorine) sampling trains, the first impinger will be enlarged to accommodate the volume of condensate expected to be collected. The concentration of the solutions for the first impinger may also be increased to address dilution due to the condensate.

Because the flue will be less than 6 inches in diameter at the location of the sampling trains, EPA Method 1A for small ducts is applicable. Because there will be no particulate in the flue gas and stratification within the duct is not anticipated, traversing of the duct during sampling will not be performed for any of the manual sampling trains. The tip of the sampling probes will be positioned in the centroid of the duct for sampling.

Because the flue gas temperature will be reduced below the range in which dioxins are formed, additional precautions are not required to avoid formation of dioxins in the sampling apparatus. If the flue gas stream is above 400 °F, dioxin formation in the sampling probe and on the filter assembly is possible. Although dioxins may not be present, if precursors are present in the sample stream, the reaction thermodynamics for dioxins formation are greatest in the temperature range of 400°F to 700°F. If the

Section: 4.0
Revision: 2
Date: 7/24/01
Page: 4-18

temperature of the gas stream is not reduced to below 300°F before exiting the sample probe, dioxins can form on particulate in the sample stream, on the walls of the sampling probe, or on the filter after the probe.

The detailed flue gas sampling procedures are presented in Appendix A.

5.0 ANALYTICAL METHODS AND CALIBRATION PROCEDURES

This section describes the process used to select the analytical methods and briefly describes methods, procedures, and calibration requirements. The frequency, acceptance criteria, and corrective actions for field calibration and QC samples are listed in Section 7.0.

5.1 SELECTION OF ANALYTICAL METHODS

The specific analytes of interest, the sample matrices, and the minimum detectable concentrations needed to achieve project objectives were considered in selecting analytical methods. The selection process involved the following general hierarchy of references:

1. EPA-approved methods described in the following references:
 - a. EPA. 1996. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Laboratory Manual, Volume 1A through 1C, and Field Manual, Volume 2. SW-846, Third Edition. Office of Solid Waste. EPA Document Control No. 955-001-00000-1. November 1986 and subsequent revisions through Revision 3, December, 1996.
 - b. 40 Code of Federal Regulations, Appendix A. 1999. *Code of Federal Regulations, Title 40, Part 60*. National Archives and Records Administration, Office of the Federal Register. Washington, DC.
 - c. EPA. 1997. Method 680 - *Toxic Polychlorinated Biphenyls by Isotope Dilution High Resolution Gas Chromatography/High Resolution Mass Spectrometry*. Office of Water. Washington, DC. Draft March 1997.
 - d. EPA. 1983. Methods for the Chemical Analysis of Water and Wastes (MCA-WW). EPA-600/4-79-020. Environmental Monitoring and Support Laboratory. Cincinnati, Ohio. Revised March 1983.
2. Other methods approved by EPA SITE for use in SITE demonstrations.

Table 5-1 summarizes the analytical methods to be performed on samples collected during the drying phase of the program. Table 5-2 summarizes the analytical methods to be performed on the samples collected during the vitrification phase of the program. All of the parameters of interest will be analyzed by EPA methods. These methods are commonly used and easily obtained; therefore, they are not discussed in detail in this section.

The PCB congener and dioxins/furans analysis will be performed by Paradigm Analytical Laboratories in Wilmington, North Carolina. All other analyses will be performed at the Kemron Environmental Services laboratory in Marietta, Ohio.

5.2 ANALYTICAL METHODS

The analytical procedures used by the laboratory will follow the referenced EPA methodologies with the exceptions and deviations noted in Appendix B. Appendix B contains analytical methods for VOCs, SVOCs, PCBs, dioxins and furans, metals including mercury, and HCl and Cl₂.

5.3 CALIBRATION PROCEDURES

Calibration procedures, the frequency of continuing calibration verification, and the criteria for evaluating the calibration data are described in the EPA analytical methods. Calibration data will be recorded in the instrument logbook and referenced to the standards preparation log to identify the source and method of preparation of the standard solutions used. Procedures to be used for instrument calibration are presented in Appendix C.

5.4 SAMPLE STORAGE AND DISPOSAL

Paradigm and Kemron will store all residual samples and sample extracts until all data has been validated and disposal of the samples is authorized by EPA. For the first 60 days after the laboratory

TABLE 5-1 ANALYTICAL METHODS FOR DRYING PHASE

Matrix and Sampling Location	Parameter	Reference Method	Name of Method
Gas (G1)	PCBs	Method 23 Method 1668	Measurement of Gaseous Organic Compound Emissions by Gas Chromatography Toxic PCBs by High Resolution Gas Chromatography/High Resolution Mass Spectrometry, GC/MS
	PCDDs, PCDFs	Method 8290	Determination of PCDDs and PCDFs from Stationary Sources by High Resolution Gas Chromatography/High Resolution Mass Spectrometry
	Metals including mercury	Method 0060/6010B/7470A	Determination of Metals in Stack Emissions/ Inductively Coupled Plasma-Atomic Emission Spectroscopy/Mercury in Liquid Waste
	SVOST	Method 0010/ 8270C	Measurement of Gaseous Organic Compound Emissions by Gas Chromatography/Mass Spectrometry
Aqueous samples (W1)	PCBs	Method 680	PCBs by Gas Chromatography/Mass Spectrometry
	Metals including mercury	Method 0060/6010B/7470A	Determination of Metals in Stack Emissions/ Inductively Coupled Plasma-Atomic Emission Spectroscopy/Mercury in Liquid Waste
Solid samples (S1, S2)	PCBs	Method 680 and 1668	Toxic PCBs by Gas Chromatography/Mass Spectrometry and Isotope Dilution High Resolution Gas Chromatography/High Resolution Mass Spectrometry
	Metals including mercury	Method 3050B/6010B/7471B	Determination of Metals in Pre- and Post- Dried Sediments. Inductively Coupled Plasma-Atomic Emission Spectroscopy/Mercury in Solid Waste
	SVOCs	Method 3540C/8270C	Determination of SVOCs in Pre- and Post-Dried Sediments

Notes: PCB - Polychlorinated biphenyl
SVOC - Semivolatile organic compound
SVOST - Semivolatile organic sampling train
Sample locations are referenced to those indicated on Figure 4-1

TABLE 5-2 ANALYTICAL METHODS for VITRIFICATION PHASE

Matrix and Sampling Location	Parameter	Reference Method	Name of Method
Gas (H, I, J)	VOST	Method 0031/ 8260B Method 5030B/8260B	Determination of VOCs
	Semi-VOST	Method 0010/ 8270C	Measurement of Gaseous Organic Compound Emissions by Gas Chromatography/Mass Spectrometry
	PCDDs/PCDFs and PCBs	Method 23 Method 8290 Method 680	Determination of PCDDs and PCDFs from Stationary Sources and Toxic PCBs by Isotope Dilution High Resolution Gas Chromatography/High Resolution Mass Spectrometry
	Hydrogen chloride and chlorine	Method 26/SW-846 Method 9057	Determination of Chlorine from Hydrogen Chloride and Chlorine Emission Sampling Train by Anion Chromatography
	Metals including mercury	Method 0060/6010B/7470A	Determination of Metals in Stack Emissions/ Inductively Coupled Plasma-Atomic Emission Spectroscopy/Mercury in Liquid Waste
Aqueous samples (E, F, G)	PCBs	Method 680	Toxic PCBs by Isotope Dilution High Resolution Gas Chromatography/High Resolution Mass Spectrometry
	Metals including mercury	Method 6010B/7470A	Inductively Coupled Plasma-Atomic Emission Spectroscopy/Mercury in Liquid Waste
	SVOCs	Method 8270C	Determination of SVOCs in Condensate and Quench Water
Solid samples (A, B, C, D,)	PCDDs/PCDFs and PCBs	Method 23 Method 680 Method 8290 Method 1668	Determination of PCDDs and PCDFs from Stationary Sources and Toxic PCBs by Isotope Dilution High Resolution Gas Chromatography/High Resolution Mass Spectrometry
	Metals including mercury	Method 6010B/7471B	Inductively Coupled Plasma-Atomic Emission Spectroscopy/Mercury in Solid Waste
	SVOCs	Method 3540C/8270C	Determination of SVOCs in Dried Sediment and Aggregate Product
	VOCs	Method 5035/8260B	Determination of VOCs

Notes: Does not include flux samples to be analyzed for PCBs, PCDDs and PCDFs, SVOCs, and metals

PCB - Polychlorinated biphenyl

SVOC - Semivolatile organic compound

PCDD - Polychlorinated dibenzo-p-dioxins

VOST - Volatile organic sampling train

PCDF - Polychlorinated dibenzofurans

Sample locations are referenced to those indicated on Figure 4-2

Section: 5.0
Revision: 2
Date: 7/24/01
Page: 5-5

receives the samples, the samples will be stored in a refrigerator at 4 °C, unless otherwise specified in the methodology. After that time, residual samples may be stored at room temperature. If not notified otherwise within 6 months after the reporting of the sample data, the laboratory may dispose of the residual samples. Samples must be disposed of in an environmentally responsible manner.

6.0 QUALITY ASSURANCE OBJECTIVES AND QUALITY CONTROL REQUIREMENTS

This section discusses QA objectives, calculation of data quality indicators, and internal QC requirements.

6.1 QUALITY ASSURANCE OBJECTIVES

The overall QA objective for this evaluation is to produce well documented data of known quality. Quality is measured by monitoring data precision and accuracy, representativeness, completeness, comparability, and reporting limits for the analytical methods. The objectives for these parameters are discussed in Sections 6.1.1 through 6.1.5.

If analytical data fail to meet the QA objectives described in this section (for example, because of matrix interferences), EPA will explain the reason in the Technology Evaluation Report (TER) and will describe the limitations and usefulness of the data in the Innovative Technology Evaluation Report (ITER). The following corrective actions may be taken for data that do not meet QA objectives: (1) verify that the analytical measurement system was in control, (2) thoroughly check all calculations, (3) use data qualifiers (flags), and (4) reprepare and reanalyze the affected samples if authorized by the EPA SITE Project Manager and if a sufficient quantity of sample is available.

6.1.1 Precision and Accuracy

Precision and accuracy goals depend on the types of samples and analyses and the ultimate use of the analytical data. Table 6-1 summarizes the precision and accuracy goals for critical parameters. Equations used to estimate precision and accuracy are given in Section 6.2.

TABLE 6-1
QA OBJECTIVES FOR CRITICAL PARAMETERS

Matrix	Compound	Analytical Method	Measure-ment Unit	Reporting Limit^b	Precision (RPD)^c	Accuracy (% Rec)^d
Sediment	PCBs	680	ng/g	1	20	75-125 MS/MSD
	Dioxins and Furans	8290	ng/kg (ppt) ^a	1	20	75-125 MS/MSD
	Mercury	7471B	mg/kg	0.001	20	75-125 MS/MSD
	SVOCs	8270C	µg/kg	660	20	75-125 MS/MSD
	Metals	6010B	mg/kg	1.0-40	20	75-125 MS/MSD
	VOCs	8260B	µg/kg	5-50	20	75-125 MS/MSD
Condensate/ Quench Water	PCBs	680	ng/L	5	20	75-125 MS/MSD
	Dioxins and Furans	8290	ng/L	1	20	75-125 MS/MSD

Notes:

- | | | | |
|---|---|---|--|
| a | Reported on a dry-weight basis | d | Accuracy as percent recovery (% Rec) |
| b | Reflects standard laboratory reporting limits for undiluted samples | e | extracts will be split and one-half held for analysis by Method 1668 pending Method 680 PCB concentrations |
| c | Precision as relative percent difference (RPD) | | |

ASTM American Society of Testing Methods
mg/L milligram per liter
ng/g nanograms per gram
ppbv part per billion volume
SPLP Synthetic precipitation leachate procedure
µg/m³ microgram per cubic meter
µg/L microgram per liter

mg/kg miligram per kilogram
ng nanaogram
ng/kg nanogram per kilogram
ppt part per trillion
µg microgram
µg/kg microgram per kilogram

TABLE 6-1 (Continued)
QA OBJECTIVES FOR CRITICAL PARAMETERS

Matrix	Compound	Analytical Method	Measurement Unit	Reporting Limit ^b	Precision (RPD) ^c	Accuracy (% Rec) ^d
Condensate/ Quench Water	Metals	6010B	mg/L	0.003-0.2	20	75-125 MS/MSD
	Mercury	7470A	mg/L	0.001	15	75-125 MS/MSD
	SVOCs	8270C	µg/L	10	20	75-125 MS/MSD
Exhaust Gas	PCBs	680 1668 (if necessary) ^e	ng	5-50	20	75-125 MS/MSD
	Dioxins and Furans	8290 and TO-9	ng	5-50	20	75-125 MS/MSD
	Metals	6010B	µg/m ³	0.001-0.05	20	75-125 MS/MSD
	Mercury	7470A	µg/m ³	0.001	20	75-125 MS/MSD

Notes:

- | | | | |
|---|---|---|--|
| a | Reported on a dry-weight basis | d | Accuracy as percent recovery (% Rec) |
| b | Reflects standard laboratory reporting limits for undiluted samples | e | extracts will be split and one-half held for analysis by Method 1668 pending Method 680 PCB concentrations |
| c | Precision as relative percent difference (RPD) | | |

ASTM American Society of Testing Methods
mg/L miligram per liter
ng/g nanograms per gram
ppbv part per billion volume
SPLP Synthetic precipitation leachate procedure
µg/m³ microgram per cubic meter
µg/L microgram per liter

mg/kg miligram per kilogram
ng nanaogram
ng/kg nanogram per kilogram
ppt part per trillion
µg microgram
µg/kg microgram per kilogram

TABLE 6-1 (Continued)
QA OBJECTIVES FOR CRITICAL PARAMETERS

Matrix	Compound	Analytical Method	Measure-ment Unit	Reporting Limit^b	Precision (RPD) ^c	Accuracy (% Rec)^d
Exhaust Gas	VOCs	8260B and TO-15	ppbv	0.04 to 0.57	20	75-125 MS/MSD
	SVOCs	8270C and TO-9	µg	50	20	75-125 MS/MSD
Aggregate Product SPLP and ASTM Extracts	PCBs	680	ng/L	5	20	75-125 MS/MSD
		1668	ng/L (ppt)	1	20	
	Dioxins and Furans	8290	ng/L (ppt)	1	20	75-125 MS/MSD
	Metals	6010B	mg/L	0.003-0.2	20	75-125 MS/MSD
	Mercury	7470A	mg/L	0.001	15	75-125 MS/MSD
	SVOCs	8270C	µg/L	10	20	75-125 MS/MSD

Notes:

a	Reported on a dry-weight basis	d	Accuracy as percent recovery (% Rec)
b	Reflects standard laboratory reporting limits for undiluted samples	e	extracts will be split and one-half held for analysis by Method 1668 pending Method 680 PCB concentrations
c	Precision as relative percent difference (RPD)		
ASTM	American Society of Testing Methods	mg/kg	miligram per kilogram
mg/L	miligram per liter	ng	nanoogram
ng/g	nanograms per gram	ng/kg	nanogram per kilogram
ppbv	part per billion volume	ppt	part per trillion
SPLP	Synthetic precipitation leachate procedure	µg	microgram
µg/m ³	microgram per cubic meter	µg/kg	microgram per kilogram
µg/L	microgram per liter		

6.1.2 Representativeness

For this project, representativeness involves sample numbers, sampling locations, and sample preparation methods. As described in Section 3.0, the evaluation plan has been designed to ensure that more than a sufficient number of samples will be collected and that each sample will be collected in a manner that ensures maximum representativeness.

6.1.3 Completeness

Completeness is an assessment of the amount of valid data obtained from a measurement system. The percent completeness is calculated by the number of samples yielding acceptable data divided by the total number of samples planned to be collected and multiplied by 100 (see Section 6.2.4). The QA objective for the degree of completeness is 100 percent. If completeness is less than 100 percent, EPA will document why this objective was not met and the potential impact of a lower percentage, if any, on the project.

6.1.4 Comparability

The comparability of the data will be maximized by using standard EPA analytical methods, when possible. All planned methods are specified, and any deviations from the methods will be documented in the TER. All results for critical parameters will be reported in standard units as shown in Table 6-1; for noncritical parameters, results will be reported in the units specified in Table 6-1 and the method. All laboratory calibrations will be based on standards traceable to the National Institute of Science and Technology (NIST). Comparability will also be maximized through the use of consistent sample collection techniques and analytical methods throughout the evaluation.

6.1.5 Reporting Limits

Expected reporting limits for critical parameters are listed in Table 6-1. These reporting limits are based on laboratory experience with the analytical method and similar matrices as well as method detection limit (MDL) studies conducted annually by the laboratory. However, these reporting limits are based on undiluted samples; actual reporting limits may be higher for samples with elevated concentrations, particularly if sample dilution is required for analysis. In addition, high moisture content can raise quantitation limits.

Reporting limits are not expected to be a concern for this project because many sample concentrations are expected to be well above a standard reporting limits. However, any results that are less than the laboratory reporting limits but greater than the method detection limits will be flagged (with a “B” or “J” flag) to indicate greater uncertainty associated with such values. In addition, the laboratory will prepare a spreadsheet containing any positive or negative results that are less than the method detection limit. These data will not be qualified or flagged but will bear a laboratory disclaimer as to the limits of the data usability.

6.2 CALCULATION OF DATA QUALITY INDICATORS

This section presents the specific calculations that will be used to describe the following data quality indicators for the critical parameters: precision, accuracy, representativeness, and completeness.

6.2.1 Precision

Precision will be estimated by analyzing duplicate MS samples or duplicate samples. The RPD between the analyte levels measured in the MS/MSD samples (or sample duplicates) will be calculated using the following equation:

$$RPD = \{ |MS - MSD| / [0.5 * (MS + MSD)] \} * 100$$

6.2.2 Accuracy

Spike sample analyses are conducted by the laboratory to assess the accuracy of specific analytical methods and to provide information on the effect of the sample matrix on the analytical methodology. Spike sample analyses are performed by adding known amounts (“spikes”) of representative target compounds to a sample aliquot that is subjected to the entire analytical procedure. The original sample (unspiked) and the spike sample results are then compared. Accuracy is reported as percent recovery (%R) of the spike, as calculated from the following equation:

$$\%R = \frac{SSR - SR}{SA} \times 100$$

Where,

%R = Percent recovery (percent)
SSR = Spike sample result (concentration units)
SR = Original sample result (concentration units) and
SA = Spike added (concentration units)

The accuracy of the analytical data is evaluated by calculating %R values for two types of spike samples: MS samples and blank spike samples. For each type of spike sample, all %R values for a given analyte are averaged to produce a single, analyte-specific average %R value, which is then compared against an established QC criterion. QC criterion may be method- or laboratory-specific QC criterion. Data qualifiers may be applied in the data validation process to certain analytical results where the %R values do not meet the established QC criteria.

6.2.3 Representativeness

Representativeness expresses the extent to which the analytical data reflect the actual media at the site. In general, the data are representative of the site conditions and characteristics. Proper sampling and

sample management procedures as documented in Section 4.0 achieve acceptable representativeness.

Representativeness will be evaluated with respect to general sample management issues, including sample documentation, preservation, handling, and transport. The evaluation of representativeness may also include a discussion of representativeness with respect to analytical-method specific issues including method deviations, presence of potential laboratory or field artifacts, indications of sample nonhomogeneity, internal standard recovery deficiencies, and surrogate recovery deficiencies.

6.2.4 Completeness

Completeness will be reported as the percentage of all measurements judged valid. The following equation will be used to determine completeness:

$$\%C = (V / T) * 100$$

Where,

%C = Percent completeness

V = Number of measurements judged valid

T = Total number of measurements

6.3 INTERNAL QUALITY CONTROL REQUIREMENTS

Internal QC consists of checks and procedures to ensure that QA objectives are met. These checks and procedures document compliance with the objectives or demonstrate the need for corrective action. Two kinds of checks are used: (1) checks monitoring field activities, such as sample collection and shipping, and (2) checks monitoring laboratory activities, such as extraction and analysis. These checks are discussed in the following sections.

6.3.1 Field Quality Control Checks

Field QC checks will be used to determine the quality of field activities, including sample collection,

handling, and shipment. In general, these QC checks will assess the representativeness of the samples and ensure that the degree to which the analytical data are representative of actual site conditions is known and documented. Field QC checks will consist of equipment blanks, field blanks, and field sample duplicates.

6.3.1.1 Equipment Blanks and Field Blanks

Equipment blanks will be used to check for potential cross-contamination of samples. Cross-contamination may occur during sampling if decontamination procedures are not adequate. Field blanks will be used to check for potential contamination of the sample by the water used to rinse equipment during decontamination. These blanks will be analyzed for PCBs and metals using the methods described previously.

The acceptance criteria for equipment blanks and field blanks will be as follows. If contamination is found in the blanks, the analytical laboratory will determine whether the contamination originated in the field or during analysis in the laboratory by preparing and analyzing a laboratory blank. Contamination is defined as any positive or negative result greater than the method detection limit. If the analytical laboratory concludes that the contamination occurred during laboratory analysis, the laboratory will implement corrective action to find the source of contamination and will reanalyze the equipment or field blank (and potentially all associated samples) if sufficient volume exists. If it is determined that contamination originated in the field, the EPA project manager will immediately be notified, and all data will be reevaluated to determine its usefulness.

6.3.1.2 Field Duplicate Samples

Field duplicate samples will be used to check the adequacy of the field homogenization procedure in distributing the contaminants of interest (PCBs and metals) uniformly in the matrix. These duplicate samples will be analyzed in the same manner as the primary samples. The analytical results of the

primary sample and the duplicate sample will be compared. If the analytical results of the duplicate sample and primary sample have an RPD within plus or minus 50 percent, field homogenization procedures will be considered adequate. However, the 30 percent RPD criterion will be considered advisory and not a QC limit.

6.3.2 Laboratory Quality Control Checks

Laboratory QC checks are designed to determine analytical precision and accuracy, demonstrate the absence of interferences and contamination from glassware and reagents, and ensure the comparability of data. Laboratory QC checks consist of laboratory control samples (LCS) and laboratory control sample duplicates (LCSD), method blank samples, MS/MSD samples, and other checks specified in the methods. The laboratory will also complete initial calibrations and continuing calibration checks.

6.3.2.1 Method Blank Samples

Method blank samples are used to identify any system and process interferences or contamination of the analytical system that may lead to reporting of elevated analyte concentrations or false positive data. The method blank samples will consist of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation, and analysis. This blank sample will undergo all of the procedures required for sample preparation. The method blank sample will be analyzed with the field samples prepared under identical conditions.

6.3.2.2 Matrix Spike/Matrix Spike Duplicates

MS/MSDs will be used to determine the accuracy and precision of the analytical results. Triplicate aliquots of the same sample are prepared in the laboratory, and each aliquot is treated exactly the same throughout the analytical method. For the MS/MSD samples, spiking compounds will be added to the aliquots at concentrations ranging from 50 percent to 100 percent of the native concentration, but not less

than 50 mg/kg (soil or solid) or 0.5 mg/L (water or leachate). Upon screening the sample chosen for MS/MSD analysis, if it is determined that the native concentration is too high for the laboratory to meet the requirement for spiking compound concentration, then the laboratory will consult with EPA before taking further action. EPA will instruct the laboratory on alternative samples for MS/MSD analysis and will consider other options for addressing the problem, including reducing the sample mass or volume used for MS/MSD analysis. Accuracy will be determined as the percent recovery of the analyte from the sample matrix. The spiking solution will be prepared using certified stock solutions. Concentrated spiking solutions will be used to minimize differences in the sample matrix resulting from dilution.

MS results outside acceptance criteria are either caused by (1) a matrix effect in the sample chosen for the spike or (2) a batch recovery that is out of control. To determine whether the observed excursion is caused by a matrix effect, LCS/LCSD samples will be analyzed (see Section 6.3.2.3). The results will indicate if matrix effect or out-of-control conditions apply. The proper corrective actions will be taken depending on these results, as indicated in the referenced methods.

6.3.2.3 Laboratory Control Spike/Laboratory Control Spike Duplicate

LCS/LCSD samples will be prepared by spiking an aliquot of organic-free water with the same solutions used for the MS/MSD samples. LCS/LCSD samples will be used to determine the accuracy and the precision of the analytical results.

7.0 FIELD AND LABORATORY DATA PROCESSING

Correct equations and procedures must be used to ensure that all data generated and processed are valid, defensible, and comparable. The following sections describe field and laboratory data processing and project data reporting for this evaluation.

7.1 FIELD DATA PROCESSING

A limited amount of field data will be generated during this project. These data will consist primarily of the operational parameters of the Minergy glass melter and the Hazen dryer, including process throughput rates, exhaust gas temperature and velocity, and any process adjustments or additives determined to be required by Minergy. All field data will initially be recorded in field notebooks, along with any associated calibration checks, assumptions, deviations, or anomalies. The data will then be transcribed to spreadsheets and evaluated as described in Section 3. All field data will be reviewed by the EPA project manager to ensure that correct data reduction procedures were utilized and that there are no transcription errors.

7.2 LABORATORY DATA PROCESSING

The following sections discuss laboratory data reduction, validation, and reporting.

7.2.1 Laboratory Data Reduction

The analytical methods to be used for this demonstration contain detailed instructions and equations for calculating compound concentrations and other parameters. Data will be reduced to the units specified in Table 6-1 using the procedures in the analytical methods. All numerical results will be reported as calculated without censoring for any laboratory reporting limits. Numerical results that are less than the reporting limit but greater than the method detection limit will be reported for all sample and QC sample

analyses; however, these data will be flagged with a “B” or “J” designation by the laboratory. In addition, the laboratory will prepare a spreadsheet containing any positive or negative results that are less than the method detection limit. These data will not be qualified or flagged but will bear a laboratory disclaimer regarding the limits of the data usability.

The analysts responsible for the measurements will enter raw data into logbooks or onto data sheets. In accordance with standard document control procedures, the laboratories will maintain on file original copies of all data sheets and logbooks containing raw data, signed and dated by the responsible analyst. Separate instrument logs will also be maintained by the laboratories to enable reconstruction of the run sequence for individual instruments. The laboratories will maintain all data on file for 5 years in a secure archive accessible only to designated laboratory personnel. The data will be disposed of in the interim only on instruction from Tetra Tech and EPA.

7.2.2 Laboratory Data Validation

Individual analysts will review the data generated each day to determine the need for corrective action or rework. Data reviewed will include calibration and QC data. Individual analysts will also review data for completeness. Data will undergo a second review process conducted usually by one of three independent reviewers (under some conditions, this second review may be conducted by an analyst that was not responsible for generating the data he or she reviewed). The team of three independent reviewers includes the laboratory supervisor. This second review is typically conducted within several days after the data are generated. Laboratory logbooks and notebooks are also reviewed on a monthly basis by the team of three reviewers.

The Tetra Tech project chemist will be responsible for post-laboratory validation of laboratory data. This will include a full review of the case narrative, QC summary forms against project requirements, and other QC criteria. In addition, a full data validation will initially be conducted on 10 percent of the

laboratory data. If significant deficiencies are identified in the 10 percent validation effort, then the entire data package will be fully validated.

Analytical outlier data are defined as QC data lying outside a specific QA objective range for precision or accuracy for a given analytical method. If QC data are outside control limits, the laboratory supervisor will investigate to determine the probable cause of the problem. If necessary, the sample will be reanalyzed, and only the reanalyzed results will be reported. If the problem is with the matrix, both initial and reanalyzed results will be reported and identified in the laboratory report. If reanalysis is not feasible, the initial analysis results will be reported, and the results will be flagged and discussed in the laboratory report.

Suspected project outlier data will initially be identified as sample data that are of questionable validity because (1) they are outside specified acceptance limits established around the central tendency estimator of the relevant data set or (2) they are otherwise grossly dissimilar from expected results. For data that are known or assumed to be normally distributed, the initial identification criterion will be the 99 percent confidence limit defined by the student's two-tailed t-distribution test. Potential outlier data will be assessed on a case-by-case basis for physical or analytical anomalies. Only if there is clear evidence that the data are of questionable validity will the data be considered project outlier data. Project outlier data will be identified and reported in the final laboratory report, but such data will not be used to determine overall project results.

7.2.3 Laboratory Reporting Requirements

A flow chart depicting the overall data handling and reporting scheme is included as Figure 7-1. The Tetra Tech project manager and project chemist will approve the completed laboratory report before it is used to prepare the ITER and TER. The laboratory will submit full analytical and QC reports to include the following, as appropriate:

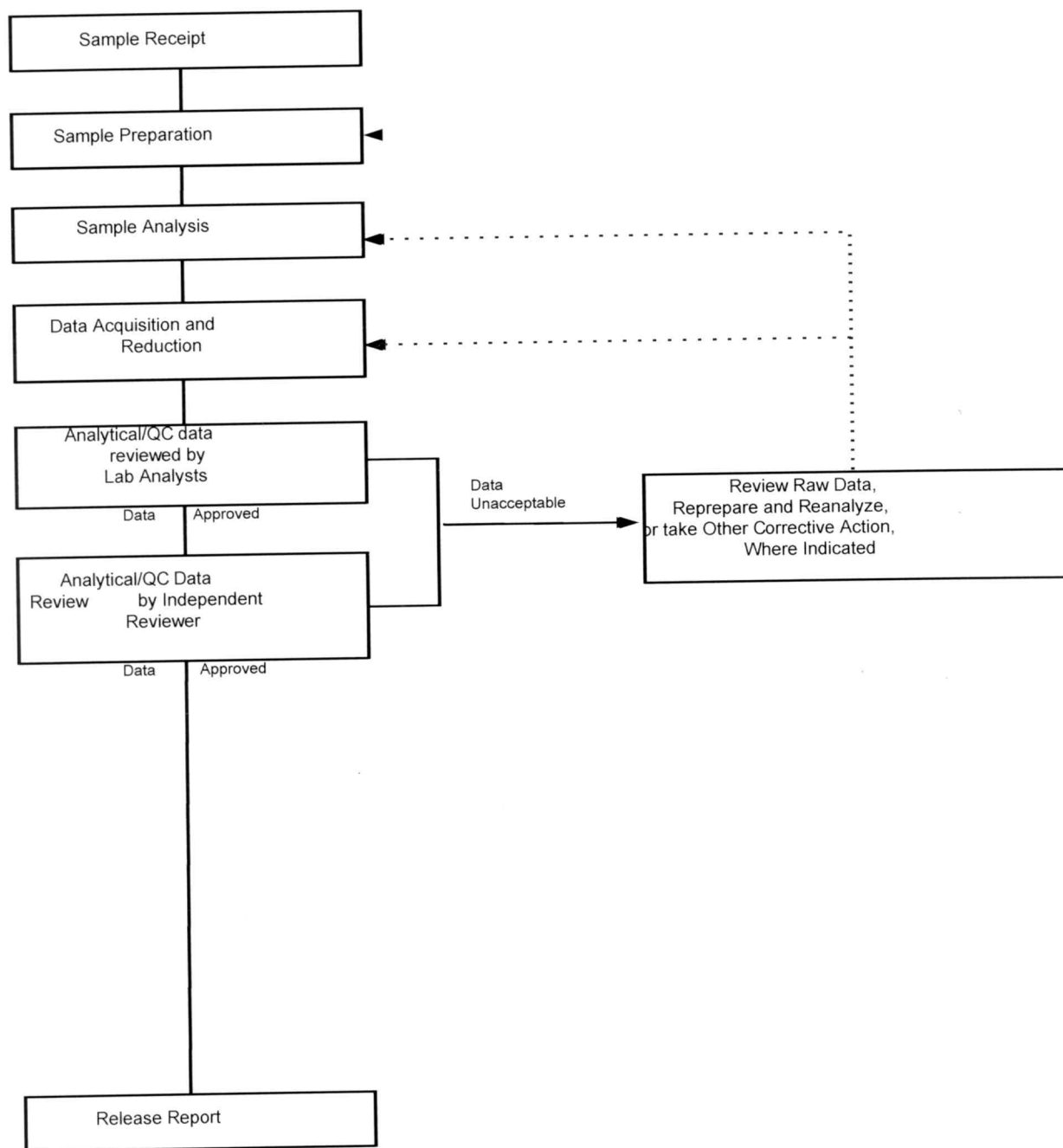


FIGURE 7-1 DATA REPORTING FLOWCHART

- Case narrative including a list of samples reviewed with field name and laboratory names cross-referenced, discussion of any deviations from the QAPP and any other nonconformances and the associated corrective actions, discussion of any analytical or procedural problems encountered and corrective actions, and an explanation of the data qualifiers used
- Completed chain-of-custody forms
- Sample result summary forms for all samples, field QC samples, and method blanks
- Spreadsheet containing any positive or negative results that are less than the method detection limit. These data will not be qualified or flagged but will bear a laboratory disclaimer as to the limits of the data usability
- QC summary forms for MS/MSD samples, LCS/LCSDs, ICP interelement correction factors, ICP linear range, and sample log-in sheets
- “True Value” summary form containing the concentrations for the ICV standard, CCV standard, CRI (CRDL) standard, ICSA solution, and ICSAB solution
- Raw data sheets and quantitation reports for initial calibration results, ICV and CCV results, ICP interference check sample (ICSA and ICSAB) results, standard results, blank results, post-digestion spike results with hand-calculated recoveries (as appropriate), and ICP serial digestion results (highlighted as such)
- Sample preparation logs and run logs In accordance with standard document control procedures, the laboratory will maintain on file original copies of all data sheets and logbooks containing raw data, signed and dated by the responsible analyst. The laboratory will maintain separate instrument logs to enable the run sequences to be reconstructed for individual instruments. The laboratory will maintain all data on file for 5 years in a secure archive warehouse accessible only to designated laboratory personnel. The data will be disposed of in the interim only after instructions to do so have been received from Tetra Tech and EPA.

7.3 PROJECT DATA REPORTING

The compilation of data from this technology evaluation project will be reported in an ITER. The ITER will contain the following information organized by section:

- 1.0 INTRODUCTION
 - 1.1 PROJECT BACKGROUND
 - 1.2 TECHNOLOGY DESCRIPTION
 - 1.3 KEY CONTACTS
- 2.0 TECHNOLOGY APPLICATIONS ANALYSIS
 - 2.1 FEASIBILITY STUDY EVALUATION CRITERIA
 - 2.1.1 Overall Protection of Human Health and the Environment
 - 2.1.2 Compliance with Applicable or Relevant and Appropriate Requirements
 - 2.1.3 Long-Term Effectiveness and Permanence
 - 2.1.4 Reduction of Toxicity, Mobility, or Volume Through Treatment
 - 2.1.5 Short-Term Effectiveness
 - 2.1.6 Ability to Implement
 - 2.1.7 Cost
 - 2.1.8 State Acceptance
 - 2.1.9 Community Acceptance
 - 2.2 TECHNOLOGY APPLICABILITY
 - 2.3 KEY FEATURES OF THE TREATMENT TECHNOLOGY
 - 2.4 MATERIALS HANDLING REQUIREMENTS
 - 2.5 SITE SUPPORT REQUIREMENTS
 - 2.6 LIMITATIONS OF THE TECHNOLOGY
- 3.0 ECONOMIC ANALYSIS
 - 3.1 INTRODUCTION
 - 3.2 CONCLUSIONS
 - 3.3 ISSUES AND ASSUMPTIONS
 - 3.4 BASIS FOR ECONOMIC ANALYSIS
 - 3.4.1 Site and Facility Preparation Cost
 - 3.4.2 Permitting and Regulatory Costs
 - 3.4.3 Equipment Costs
 - 3.4.4 Start-up and Fixed Costs
 - 3.4.5 Labor Costs
 - 3.4.6 Consumables and Supplies Cost
 - 3.4.7 Utilities Costs
 - 3.4.8 Effluent Treatment and Disposal Costs
 - 3.4.9 Residuals and Waste Shipping, Handling, and Transport Costs
 - 3.4.10 Analytical Costs
 - 3.4.11 Facility Modifications, Repair, and Replacement Costs
 - 3.4.12 Site Restoration Costs
- 4.0 TREATMENT EFFECTIVENESS
 - 4.1 BACKGROUND

4.1.1	Site Description	
4.1.2	Evaluation Objectives and Approach	
4.2	EVALUATION PROCEDURES	
4.2.1	Evaluation Preparation	
4.2.2	Evaluation Design	
4.2.3	Sampling Methods	
4.2.3.1	PCB/Dioxins/Furans Analysis	
4.2.3.2	Metals including Mercury	
4.2.3.3	SVOC Analysis	
4.2.4	Quality Assurance and Quality Control Program	
4.2.4.1	Field Quality Control Program	
4.2.4.2	Laboratory Quality Control Checks	
4.2.4.3	Field and Laboratory Audits	
4.3	EVALUATION RESULTS AND CONCLUSIONS	
4.3.1	Primary Objectives	
4.3.2	Secondary Objectives	
4.3.3	Data Quality	
4.3.4	Conclusions	
5.0	TECHNOLOGY STATUS	
6.0	REFERENCES	
APPENDIX A - VENDOR CLAIMS		
APPENDIX B - CASE STUDIES		

8.0 ASSESSMENTS

Assessments of measurement systems and associated data will be performed on both a day-to-day basis by Tetra Tech project staff (“routine assessments”), and as directed, rigorous investigations by independent technical experts (“audits”). This section discusses QA audits, corrective actions, and QA reports to management.

8.1 QUALITY ASSURANCE AUDITS

On a routine basis, the entire EPA project team, including the Tetra Tech project manager, field staff, QA staff, and subcontracted laboratories, will assess measurement systems and identify data quality issues as they arise. Identification of data quality issues will be based on the following:

- Assessments of equipment adequacy or condition for the intended measurement
- ◻ Any difficulties involved in implementing the sampling and analytical methods identified in the QAPP
- ◻ Comparison of actual results to what is reasonably expected
- ◻ Evaluation of the internal consistency of results
- ◻ Comparison of quality control results to acceptance criteria or QA objectives
- ◻ Any other evidence uncovered during day-to-day operations that measurement systems are not in control or that data are of questionable validity.

Routine assessments are often the most effective in identifying data quality issues as they arise.

However, personnel directly and intimately involved in a project may not always recognize when a data quality issue has arisen. Therefore, audits will be conducted (1) to provide an independent overview of measurement systems and data and (2) to provide additional assurance that data quality issues are identified and appropriately corrected.

8.1.1 Internal Audits

QA audits are independent assessments of measurement systems. QA audits may be internal or external and may involve performance or systems reviews. Tetra Tech personnel who are independent of the sampling and analytical teams conduct internal audits. An independent organization, typically EPA, conducts external audits.

For this project, an internal technical systems audit (TSA) of field sampling and measurements systems is planned during the Minergy melter demonstrations. At a minimum, the following activities will be audited during the field TSA:

- C Sample collection
- C Process measurements
- C Field QA/QC
- C Field documentation
- C Decontamination
- C Sample labeling, packaging, and shipping
- C Project management/QA management of quality-affecting activities

Tetra Tech also will conduct a TSA of Pardigm analytical laboratories and its implementation of the analytical methods. In addition, the TSA will include the following at a minimum:

- C Sample receipt/sample storage
- C Internal chain-of-custody procedures
- C Sample extraction and digestion
- C Sample analysis

- C Standards preparation, storage, and use of second source standards
- C Calibration
- C QC
- C Data reduction, validation, and reporting
- C Project management/QA management of quality-affecting activities

All field and laboratory TSAs performed by Tetra Tech will be conducted by a technical expert designated by the Tetra Tech Site QA manager. The Tetra Tech Site QA manager will ensure that each auditor is sufficiently removed organizationally from project activities to provide an independent assessment. Auditors may be either Tetra Tech employees or consultants and will be assigned prior to the initiation of measurement activities.

The TSAs will be conducted in accordance with internal guidance for audits under the EPA SITE program and in accordance with EPA/SITE technical directives and guidance. Based on internal guidance, the audit process to be implemented by the assigned auditor can be summarized as the following sequential steps:

1. A checklist is developed from the EPA-approved QAPP and from reference methods identified in the QAPP.
2. Actual project activities are observed and compared to the activities described in the EPA-approved QAPP and in the prescribed reference methods using the prepared checklist.
3. Nonconformances and corrective actions are discussed on site; any immediate corrective action is observed and documented when possible.
4. A draft TSA report is prepared documenting any observed nonconformances as well as any immediate corrective action that was implemented.
5. The draft TSA report is subjected to technical, editorial, and QC reviews.

6. The draft TSA report is distributed to the auditee, the EPA/SITE Project Manager, the EPA/SITE Divisional QA Manager, and the Tetra Tech project manager and SITE QA manager.
7. Any response to the draft TSA report is reviewed to assess the issue or proposed corrective action.
8. A final TSA report is prepared, subjected to internal review, and distributed to the auditee, the EPA work assignment manager and Divisional QA Manager, and the Tetra Tech project manager, SITE program manager and SITE QA Manager.

8.1.2 External Audits

TSAs of sampling and analysis procedures may also be conducted by EPA or a designated EPA contractor, at the discretion of the EPA work assignment manager and divisional QA manager. Most commonly, EPA will audit Tetra Tech's field sampling and measurement activities. If EPA elects to perform a field audit, Tetra Tech will endeavor to coordinate the above-described internal TSA with the EPA audit and to schedule the audits on sequential days. The internal TSA will then be identified as a pre-audit and will be used to identify issues for resolution during the EPA audit. If EPA does not elect to perform a field audit, Tetra Tech will include the EPA work assignment manager and divisional QA manager in the debriefing for the internal TSA and will provide all documentation from the internal TSA to EPA for review.

Performance audits of critical analytical procedures may also be conducted by Tetra Tech or by EPA by using blind QC samples that incorporate selected critical analytes or representative compounds for analysis by the subcontracted laboratory. However, no performance audits are currently planned for this project by Tetra Tech.

8.2 CORRECTIVE ACTIONS

Each member of the EPA project team is responsible for initiating a nonconformance memorandum whenever any field or laboratory measurement activity is either not in compliance with the EPA-

approved QAPP or demonstrates a potential data quality issue during routine assessments. The nonconformance memorandum should document the problem, the probable impact on the quality of the associated data, and the immediate corrective actions implemented. A copy of all nonconformance memoranda initiated by the field team will be forwarded to the Tetra Tech project manager to ensure that appropriate corrective action is implemented. All nonconformance memoranda initiated by laboratory personnel will initially be forwarded to the laboratory project manager, who will determine whether the nonconformance has been corrected appropriately and whether consultation with the Tetra Tech project manager is needed (the Tetra Tech project manager should be consulted for all nonconformances that could significantly impact project data). The Tetra Tech project manager will consult with the Tetra Tech SITE QA manager, who will review issues with the EPA divisional QA manager, as needed, to develop an appropriate plan of corrective action.

Once the corrective action to be implemented has been identified, a corrective action memorandum will be initiated by the Tetra Tech project manager or by the laboratory project manager, laboratory supervisor, or laboratory QA manager. The corrective action memorandum will document the corrective action ultimately decided upon, who was involved in the decision-making, and who is responsible for implementing the corrective action.

The Tetra Tech project manager will retain a copy of all nonconformance and corrective action memoranda generated by Tetra Tech project staff. A copy of all laboratory nonconformance memoranda and corrective action memoranda will be retained by the laboratory project manager and then included in the laboratory's final data report.

For field nonconformances, the Tetra Tech project manager is directly responsible for ensuring that appropriate corrective action has been taken. For laboratory nonconformances, the laboratory project manager, laboratory supervisor, and laboratory QA manager are responsible for assuring that appropriate corrective action has been implemented and for keeping the Tetra Tech project manager informed of the status of laboratory corrective actions.

For corrective actions taken in response to audits, the audit report and response serves as the documentation of the nonconformance and of the corrective action taken. The Tetra Tech project manager and laboratory manager are responsible for ensuring that corrective actions identified through the audit process are completely implemented for field and laboratory activities, respectively.

8.3 QUALITY ASSURANCE REPORTS TO MANAGEMENT

Effective management of environmental measurement efforts requires timely assessment and review, facilitated by effective interaction and feedback among team members. Periodic internal reports are necessary to provide ongoing evaluation of measurement data quality. Such reports may include the following:

- C A summary of project activities and general QA program status
- C A summary of any technology evaluation plan changes
- C A summary of unscheduled maintenance
- C A summary of corrective actions
- C Monthly reports indicating the status of any unresolved problems

The final laboratory report for this project will include a separate QA section documenting QA/QC activities that lend support to the credibility of the data and the validity of the conclusions. The QA section will include the following items, as appropriate:

- C Changes to the QAPP
- C Limitations or constraints on the applicability of the data
- C Results of QA audits
- C Assessments of data quality in terms of precision, accuracy, completeness, detection limits, representativeness, and comparability

9.0 REFERENCES

- Minergy Corp. 2001. E-Mail Message Regarding Sediment Mixing. From Terry Carroll, Project Contact. To Joe Dauchy, Ph.D., Environmental Scientist, Tetra Tech EM, Inc. January 12.
- Minergy Corp. 2001. E-Mail Message Regarding QAPP. From Terry Carroll, Project Contact. To Ken Brown, Environmental Scientist, Tetra Tech EM, Inc. February 12.
- Tetra Tech EM, Inc. 2001. E-Mail Message Regarding Observation Notes from Oversight of Sediment Drum Sampling. From Jane Mari. To Joe Dauchy, Ph.D., Environmental Scientist, Tetra Tech EM, Inc. January 10.
- Tetra Tech EM, Inc. 2001. E-Mail Message Regarding Observation Notes from Oversight of Soil Homogenization. From Jane Mari. To Joe Dauchy, Ph.D., Environmental Scientist, Tetra Tech EM, Inc. January 18.
- U.S. Environmental Protection Agency. 1991. "Preparation Aids for the Development of Category II Quality Assurance Project Plans. Publication No. EPA/600/8-91/004. August.
- U.S. Environmental Protection Agency. 1992. "Specifications and Guidance for Obtaining Contaminant-Free Sampling Containers." OSWER Directive No. 9240-O5A. April.
- U.S. Environmental Protection Agency. 1998. "QAPP Requirements for Applied Research Projects". National Risk Management Research Laboratory. August 18.
- Wisconsin Department of Natural Resources. 1999. Remedial Investigation / Feasibility Study.
- Wisconsin Department of Natural Resources. 2001. Wisconsin Administrative Code, Chapter NR 140. Groundwater Quality. April.
- Wisconsin Department of Natural Resources. 1997. Wisconsin Administrative Code, Chapter NR 538. Beneficial Reuse of Industrial Byproducts. December.

APPENDIX A

DETAILED FLUE GAS SAMPLING PROCEDURES

Appendix A

Detailed Flue Gas Sampling Procedures

Details of the sample trains, sampling procedures, and recovery procedures are discussed in the subsequent sections of this appendix.

A.1 EPA Methods 1, 1A ,2, and 2A--Traverse Point Determination, Stack Gas Temperature, Velocity, and Volumetric Flow Rate

EPA Method 1 or 1A will be used to determine the sample and velocity traverse points for velocity measurements and isokinetic sampling. With EPA Method 1, the duct or stack cross-section is divided into equal areas. A traverse point is located in each of the resulting areas. The minimum number of equal areas and traverse points depends on the duct diameter and length in equivalent diameters directly upstream and downstream of the sample location. Schematic layouts of the sample locations and traverse points are not available during the writing of this test plan. Schematics will be created and presented in the ensuing report for this program. Application of Method 1 includes additional considerations for obtaining high quality test results. Method 1A is identical to Method 1 in principle, but the applicability is limited to ducts from 4- to 12-inches in diameter. If the sampling ports are located in ducts smaller than 4 inches in diameter, the duct will not be traversed. The tip of the probe will be positioned in the centroid of the duct for sampling. Traversing of the duct will not be performed because the flow characteristics of the flue gas stream will be changed by the flow disturbance created by the sampling probe and the port (through which the probe is inserted into the duct).

The sample location may have acceptable geometry but will still exhibit swirling, stratification, or asymmetrical flow, which may reduce the quality of the measurements. The acceptable limit for average swirl of the flue gas is 20°. Where swirl exceeds this limit, the method recommends that either modifications to the duct be made to straighten the flow or an alternate sample location be chosen. The presence of nearby elbows or dampers may cause this type of highly

asymmetrical flow profiles. Sample locations should also be reconsidered when the velocity difference at different traverse points exceeds 50 percent of the average, locations where the average velocity head is less than 0.05 inches water, or where the velocity is less than 10 feet per second; at such low velocity heads, the S-type pitot calibration coefficient is less reliable. If the stack or duct to be sampled do not meet the minimum dimension requirements of Method 1 to conduct a traverse, then the nozzle of the sampling probes will be located in the centroid of the duct, and no traversing will be performed during sample collection.

EPA Method 2 and Method 2A will be used to determine the stack gas temperature, velocity, and volumetric flow rate. The velocity of the stack gas is determined from the density of the gas and the measurement of the average velocity head. A stainless-steel sheathed Type K thermocouple (TC) is used to measure stack temperature; a stainless-steel Type S pitot and an incline manometer of applicable sensitivity are used to measure stack gas velocity. To minimize the mutual interference, the TC and pitot will be assembled according to the method specifications. Pre-test and post-test leak checks will be conducted to ensure the accuracy of the velocity measurements.

For each test, an initial pre-test velocity traverse will be performed to obtain preliminary temperature, velocity, swirl, and other critical source information. This information will be used to verify the suitability of the sample location, as well as provide pre-test data for sizing isokinetic sampling nozzles and setting other necessary run parameters. For the isokinetic and nonisokinetic sample trains, the velocity will be measured concurrently with the sample collection. The averages of the pre-test and post-test velocities and flow rates will be used for the sampling periods.

A.2 EPA Methods 3A and 4 -- Stack Gas Analysis and Moisture Content Determination

EPA Method 3A will be used to determine the stack gas oxygen and carbon dioxide concentrations and dry molecular weight. A stack gas sample will be collected from the stack and examined using instrumental methods to determine carbon dioxide and oxygen content. The dry molecular weight of the stack gas will be calculated using the measured oxygen and carbon dioxide levels, assuming the remainder of the stack gas composition is nitrogen. Low levels (in the parts per million range) of carbon monoxide, sulfur dioxide, nitrogen oxides, hydrocarbons, and other compounds are not significant factors in the molecular weight determination. The molecular weight and excess oxygen levels are used in velocity, isokinetic sampling rate, and pollutant emission rate calculations.

EPA Method 4 is normally used to determine the moisture content of the stack gas. This method is applicable for a pre-test determination of moisture content or when a moisture determination will not be made as part of the other methods. For this study, moisture will be determined using a modified impinger train based on the EPA Method 0010 train.

As flue gas is drawn through the sample train, moisture in the gas condenses and is collected in the impingers. The amount of water collected in the impinger train is determined gravimetrically. The moisture content is determined from the amount of water collected and the amount of flue gas sampled. The wet molecular weight of the flue gas is calculated from the flue gas moisture content and the dry molecular weight. Due to the design of the combustion control system, the potential exists for saturation of the flue gas and the presence of entrained water droplets. Moisture content calculated from the Method 0010 train will be compared to the psychrometric charts to determine if saturation has occurred.

A.3 EPA Method 0010 -- Semivolatile Organic Compounds

SVOCs will be collected using EPA Method 0010 during both the drying phase and the melting phase of the program. The resulting sample fractions will be shipped or transported to the analytical laboratory for preparation by Method 3542 and analysis by Method 8270C. The Method 0010 sample train configuration is adapted from EPA Method 5 procedures, and is therefore referenced as Modified Method 5. In addition to the Method 5 sampling train components, a condenser coil and a sorbent module containing a porous polymeric resin (XAD-2) will also be used. The XAD-2 module is used to collect SVOCs that pass through the high efficiency glass or quartz fiber filter in the gas phase. An optional heated Teflon® sample transfer line may be used between the filter box and condenser coil, if the sampling platform or sampling port configuration do not allow proper orientation of the sorbent module. The filter will be used to collect organic-laden particulate materials; however, particulate matter is not anticipated to be present in the flue from either of the processes being sampled. Gaseous and particulate pollutants will be withdrawn from the emission source in an isokinetic manner and collected in the sampling train illustrated in Figure A-1. Because the diameter of the ducts in both the dryer and the melter will be less than 12 inches in diameter, sampling at an isokinetic rate may not be possible. An overview of the sampling method is shown in Figure A-2. If the sampling duct does not meet the minimum stack dimension requirements as noted in Method 1, then traversing of the duct will not be performed.

There are a number of possible interferences with this sampling procedure. Reaction of nitrogen oxides in the determination of certain water soluble compounds, such as phenol and urethane, will reduce their measured concentration. Other possibilities that could result in positive or negative bias include the following: (1) stability of the extracted compounds in methylene chloride; (2) the formation of water-soluble organic salts on the resin in the presence of moisture;

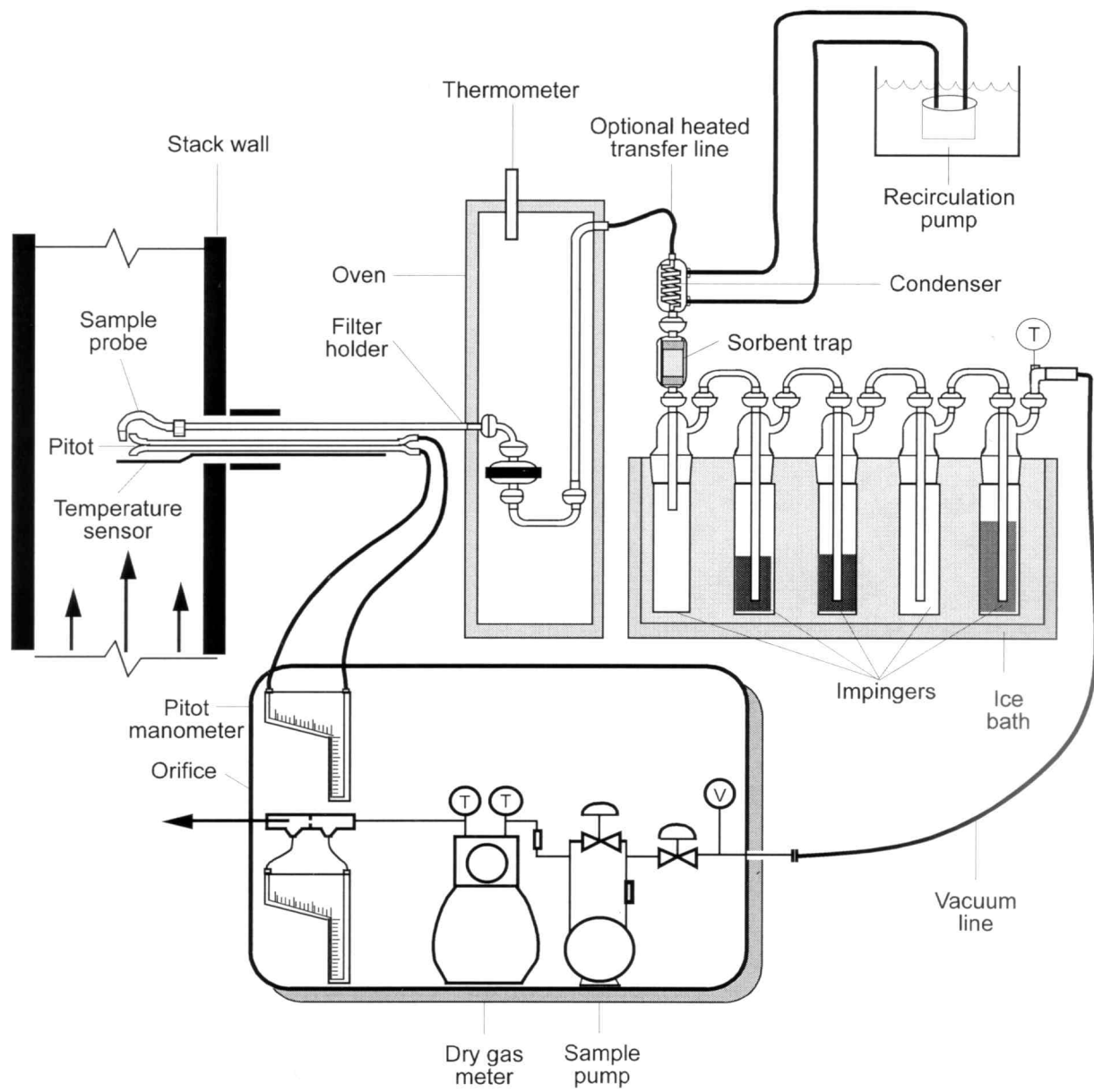


FIGURE A-1 METHOD 0010 SEMIVOLATILE SAMPLING TRAIN SCHEMATIC

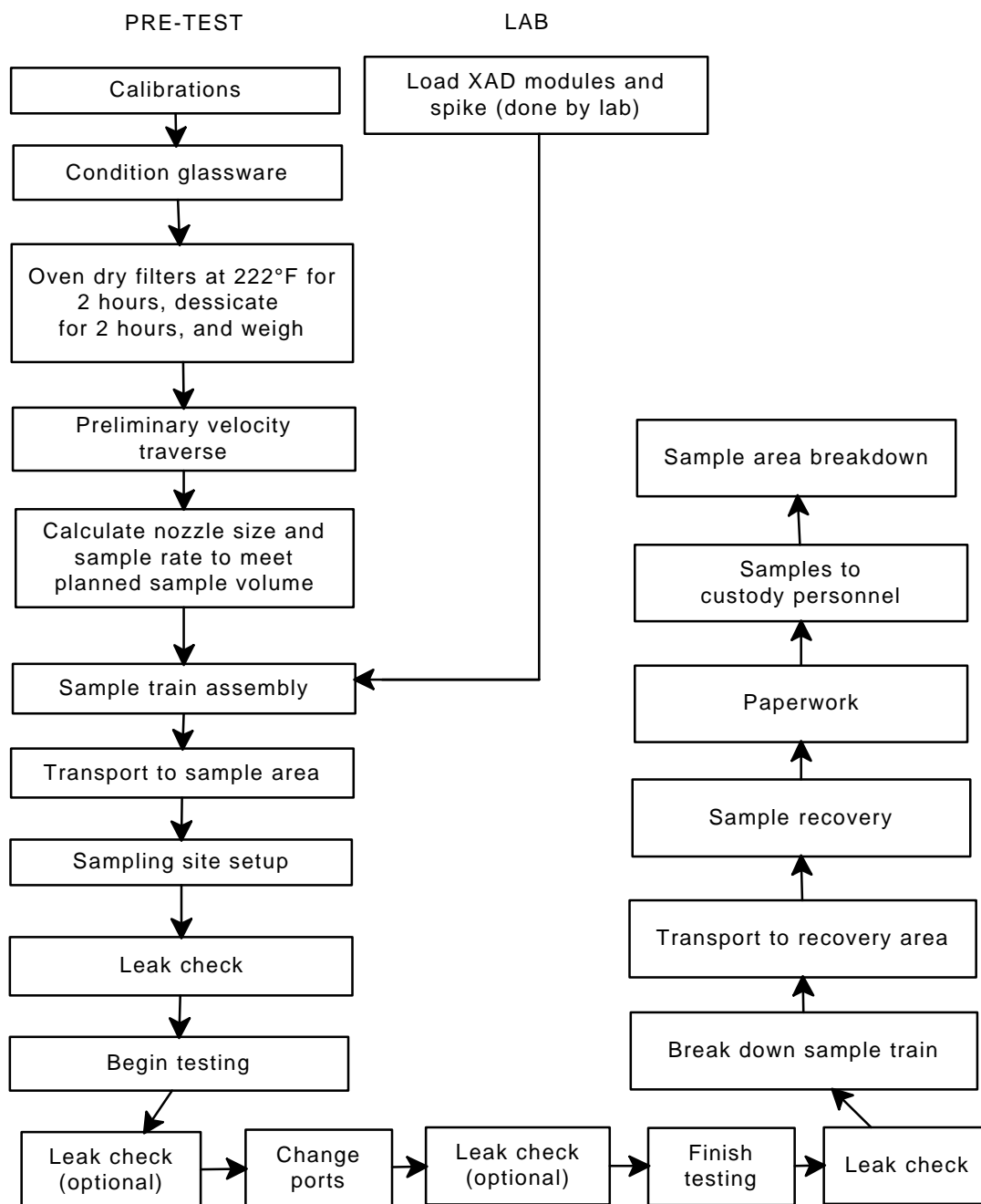


FIGURE A-2

METHOD 0010 OVERVIEW

(3) the solvent extraction efficiency of water-soluble compounds from aqueous media; and (4) photodegradation of absorbed organic compounds on the resin. To reduce the chance of sample contamination, all glassware will be thoroughly cleaned before use (see Figure A-3). All glassware and other components (such as Teflon[®]-coated filter supports, tweezers, Teflon[®] squeeze bottles, and probe brushes) will first be washed in hot water with a non-phosphorus laboratory detergent, rinsed with tap water, and then rinsed with deionized water. All glassware, excluding volumetric glassware, will then be baked at 450 °C for at least 1 hour. Finally, all glass components will be rinsed with a series of solvent washes and dried in an organic, solvent-free area. After cleaning, components will be sealed with glass caps and Teflon[®] tape. All sample recovery containers will be purchased as “certified,” indicating the containers have been cleaned to meet EPA Contract Laboratory Program analyte specifications (EPA 1992). Sampling media will be prepared as specified in the methodology by the analytical laboratory.

Assembly of the SVOC sampling train will begin in the recovery area. Table A-1 lists the equipment needed to assemble the sampling train. Table A-2 lists the equipment required to recover the sampling train. During the assembly of the train, a visual inspection of each of the sampling train components and equipment will be performed. Glassware will be checked for cracks and chips, and all connections will be cleaned to ensure good seals. Fittings and ferrules will be inspected for wear and tubing will be inspected for wear and flexibility. If any components are damaged or defective, the component will be replaced. All openings of the train will be covered with aluminum foil or Teflon[®] tape during the assembly process. No silicone grease will be used on any ground glass joints prior to the organic resin module.

The probe will be constructed of a single, heated, quartz glass tube, bent at the nozzle. The temperature of the probe will be monitored by a thermocouple placed under the probe heater in the back half of the probe. The nozzle is tapered to meet sharp-edged nozzle requirements.

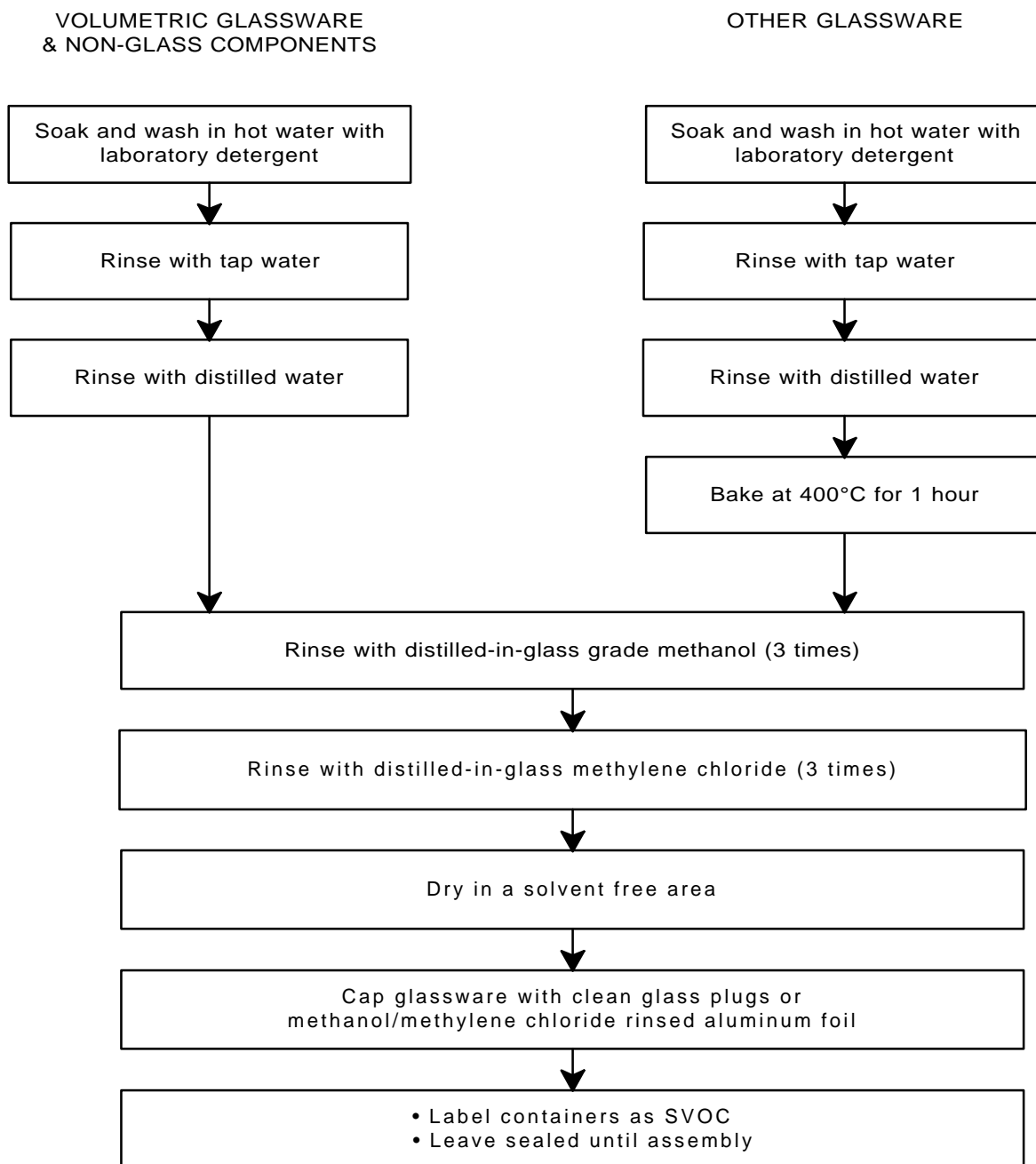


FIGURE A-3 METHOD 0010 - GLASSWARE PREPARATION PROCEDURE

TABLE A-1 SAMPLE TRAIN COMPONENT CHECKLIST FOR METHOD 0010

Probe nozzle	- Stainless steel (316) or quartz
Probe liner	- Borosilicate glass or quartz tubing
Probe sheath and heater	- Stainless steel (316) probe sheath capable of maintaining the exit or the probe at $120^{\circ}\text{C} \pm 12^{\circ}\text{C}$
Pitot tube	- Type S (as per EPA Method 2)
Differential pressure gauge	- Inclined manometer (as per EPA Method 2)
Filter holder	- Borosilicate glass with a glass frit filter support and gasket
Filter heating system	- Capable of maintaining the filter holder at $120^{\circ}\text{C} \pm 12^{\circ}\text{C}$
Organic sampling module	- Unit consists of a gas-conditioning section, a sorbent trap, and a condensate knockout trap sized to remove moisture from sample
Water circulating bath	- Bath and pump capable of circulating water to the condenser and sorbent trap water jackets
Impinger train	- Three 500-milliliter (ml) impingers, connected in series with leak-free ground-glass joints
Metering system	- Vacuum gauge, leak-free pump, thermometers, dry gas meter, and related equipment, as shown in Figure 4-1
Barometer	- Aneroid, or other barometer capable of measuring to within 2.5 millimeter mercury (mm Hg)
Gas density equipment	- Temperature sensor, pressure gauge, and gas analyzer
Calibration and field records	- A permanently bound laboratory notebook

TABLE A-2 SAMPLE RECOVERY CHECKLIST FOR METHOD 0010

Probe liner brush	- Nylon bristle brush with stainless-steel wire handle, extensions made of an inert material at least as long as the probe
Wash bottles	- Teflon® or glass (3)
Sample storage containers	- Chemically resistant, borosilicate amber and clear glass with screw-cap liners made of Teflon® (1,000 ml, 500 ml, 250 ml)
Petri dishes	- Glass, sealed with Teflon® tape for transportation of samples
Graduated cylinder/balance	- To measure condensed water to nearest 1 ml or 1 gram (g)
Plastic storage containers	- Screw-cap polypropylene or polyethylene for silica gel
Funnel and rubber policeman	- To aid in the transfer of silica gel
Funnels	- Glass, to aid in sample recovery
Teflon® tweezers	- For filter recovery
Filters	- Glass or quartz-fiber, without organic binder, 99.95 percent efficient
Crushed ice	- For cooling train during the run and for transport of samples
Stopcock grease	- Solvent-insoluble, heat-stable silicone grease
Glass wool	- Used to plug the unfritted end of the sorbent module
Adsorbent resin	- Porous polymeric resin (XAD-2 or equivalent), cleaned prior use
Silica gel	- Indicating type, 6 to 16 mesh
Impinger solutions	- Distilled organic-free water (Type II) 1Normal solution of sodium hydroxide (NaOH)
Methylene chloride	- Distilled-in-glass grade
Methanol	- Distilled-in-glass grade
Water	- Distilled organic-free (Type II)

During the melting phase of the program, if the temperature of the flue is greater than 900 °F, an air-cooled probe may be used, where the outer sheath of the probe is jacketed with a flow of compressed air. This allows the interior probe liner to be maintained at a constant temperature without absorbing the radiant heat from the flue. The use of the air-cooled probe is not anticipated. The probe will be constructed of a single, heated, quartz glass tube, bent at the nozzle. The temperature of the probe will be monitored by a thermocouple placed under the probe heater in the back half of the probe. The nozzle is tapered to meet sharp-edged nozzle requirements. During the melting phase of the program, if the temperature of the flue is greater than 900 °F, an air-cooled probe may be used, where the outer sheath of the probe is jacketed with a flow of compressed air. This allows the interior probe liner to be maintained at a constant temperature without absorbing the radiant heat from the flue. The use of the air-cooled probe is not anticipated. The temperature of the optional heated transfer line will be monitored by a thermocouple inserted between the Teflon[®] tube and the heating jacket near the probe-end of the line (if the heated transfer line is used). The filter assembly will be installed in a temperature-controlled oven to maintain a temperature of 248 °F ± 25 °F. The filter assembly is constructed of borosilicate glass, and the filter support is constructed of Teflon[®]. A water-cooled condenser is located downstream of the heated filter oven. The condenser is followed by a water-cooled sorbent module containing about 50 g of precleaned XAD-2 resin. The analytical laboratory will prepare and load the XAD-2 resin into the absorbent cartridges according to Method 0010. The laboratory will supply filters of the appropriate size to use in the sampling train. The filters will be cleaned to remove any organic matter. The filters must exhibit at least 99.95 percent RE on 0.3 micrometer (µm) dioctyl phthalate smoke particles (typically vendor documentation of this is sufficient). During sampling, the XAD-2 cartridge inlet temperature will be monitored to ensure that the temperature of the flue gas sample entering the module is maintained below 20°C to ensure efficient capture of organic compounds.

The XAD-2 resin module is located above an empty impinger. The impinger is used to collect any condensate in the flue gas sample. The first impinger is modified without a stem so the sample gas does not bubble through the collected condensate. An additional empty impinger may be added if the stack moisture conditions yield a large volume of condensate. The second impinger in the sampling train is of Greenberg-Smith (GS) design. The third and fourth impingers will be of modified GS design. The second impinger will contain 100 ml of organic free Type II water. The third impinger will be charged with 1 N sodium hydroxide to scrub any hydrogen chloride and sulfur dioxide from the sample stream prior to the pump. The fourth will contain a known weight of silica gel. Impinger weights and contents will be recorded on an impinger weigh sheet, which stays with the impinger set during the test. All connections within the train will be glass or Teflon®. The impingers are followed by a meter box containing a pump, dry gas meter, and a calibrated orifice meter. Assembly procedures are shown in Figure A-4.

Following completion of the organics train pre-test checklist, the operator informs the crew chief and awaits instructions to position the probe at the first traverse point (if traversing is required) and begin sampling. Stack gas velocity is measured at each traverse sampling point with an s-type pitot probe. If the duct diameter is less than 12 inches, the duct will not be traversed, and the probe tip will be located in the centroid of the duct. A hand calculator is used to calculate the orifice meter reading corresponding to isokinetic flow. All field data will be recorded using black ballpoint pens.

The sampling train will be leak checked at the sampling site, after the train has reached sampling temperature and prior to any sampling activities. The sampling pump will be started with the fine adjustment completely open and the course adjustments completely closed. The coarse adjustment will be opened slowly while the fine adjustment is being closed until a vacuum of 15 mm Hg is reached. A leak rate of no more than 0.00057 cubic meters per minute (m^3/min) or

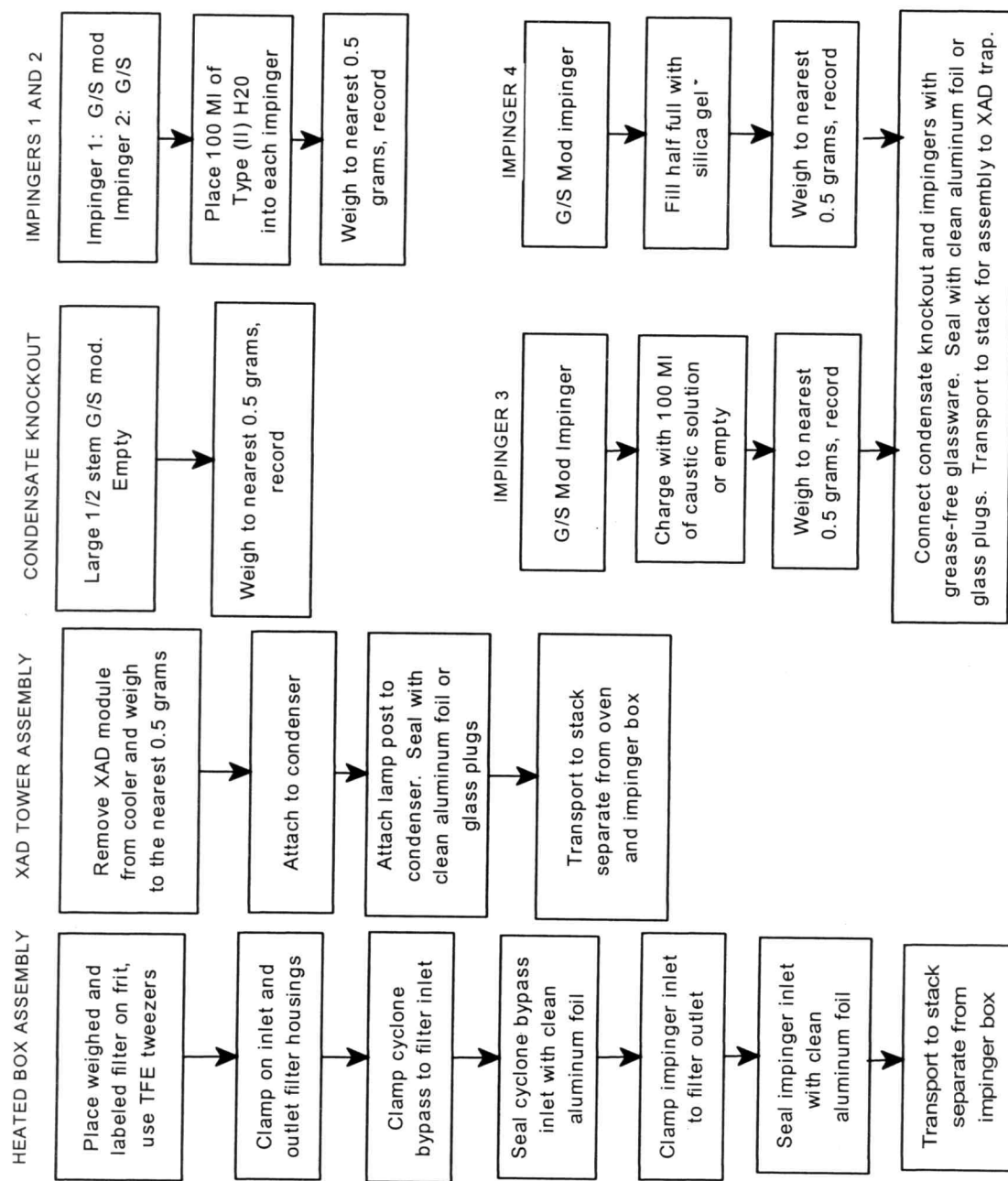


FIGURE A-4

METHOD 0010 SAMPLING TRAIN ASSEMBLY PROCEDURE

0.02 cubic feet per minute (cfm) or 4 percent of the sampling rate, whichever is less. Leak check procedures are shown in Figure A-5.

When sampling has been completed, the sample train will be partially disassembled, as shown in Figure A-6, and delivered to the recovery area. The probe and flexible line are then rinsed with the specified solvents into a socket flask attached directly to the end of the probe or line. This approach reduces the possibility of sample loss during recovery. The recovery containers for liquids will be contaminant-free 1,000- or 500- ml amber glass bottles with Teflon[®]-lined lids. Glass petri dishes will be used for filter storage. XAD-2 resin modules will be capped with prewashed glass plugs. Following recovery, the sample train will be reassembled with a fresh XAD-2 cartridge, and all openings will be sealed with prewashed glass plugs. Figure A-7 shows the recovery scheme to be used for this test program.

A.4 EPA METHOD 23 (modified) - DIOXIN, FURAN, PCB EMISSIONS

Polychlorinated dibenzo-p-dioxins (PCDD), polychlorinated dibenzofurans (PCDF), and PCBs will be collected using EPA Method 23 during the vitrification phase of the program. Method TO-4A/TO-9A was substituted for Method 23 for the dryer demonstration. The laboratory will extract the components of the sampling train using Method 23 and will analyze the resulting extract using Method 680 for PCBs. The Method 23 sample train configuration is adapted from EPA Method 5 procedures.

In addition to the Method 5 sampling train components, a condenser coil and a sorbent module containing a porous polymeric resin (XAD-2) will also be used. The XAD-2 module is used to collect SVOC material that pass through the high efficiency glass or quartz fiber filter in the gas phase. An optional heated Teflon[®] sample transfer line may be used between the filter box and

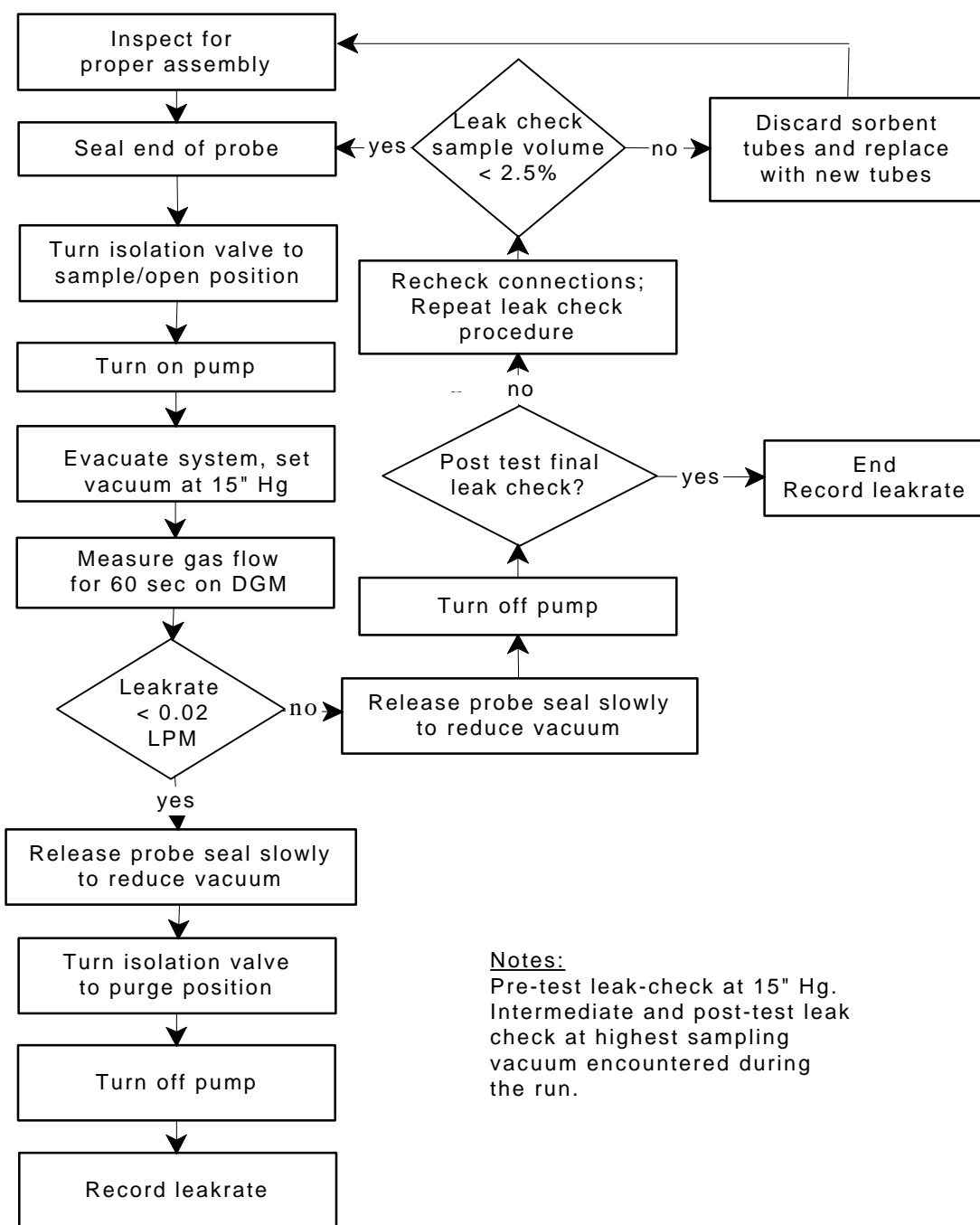


FIGURE A-5

METHOD 0010 LEAK CHECK PROCEDURE

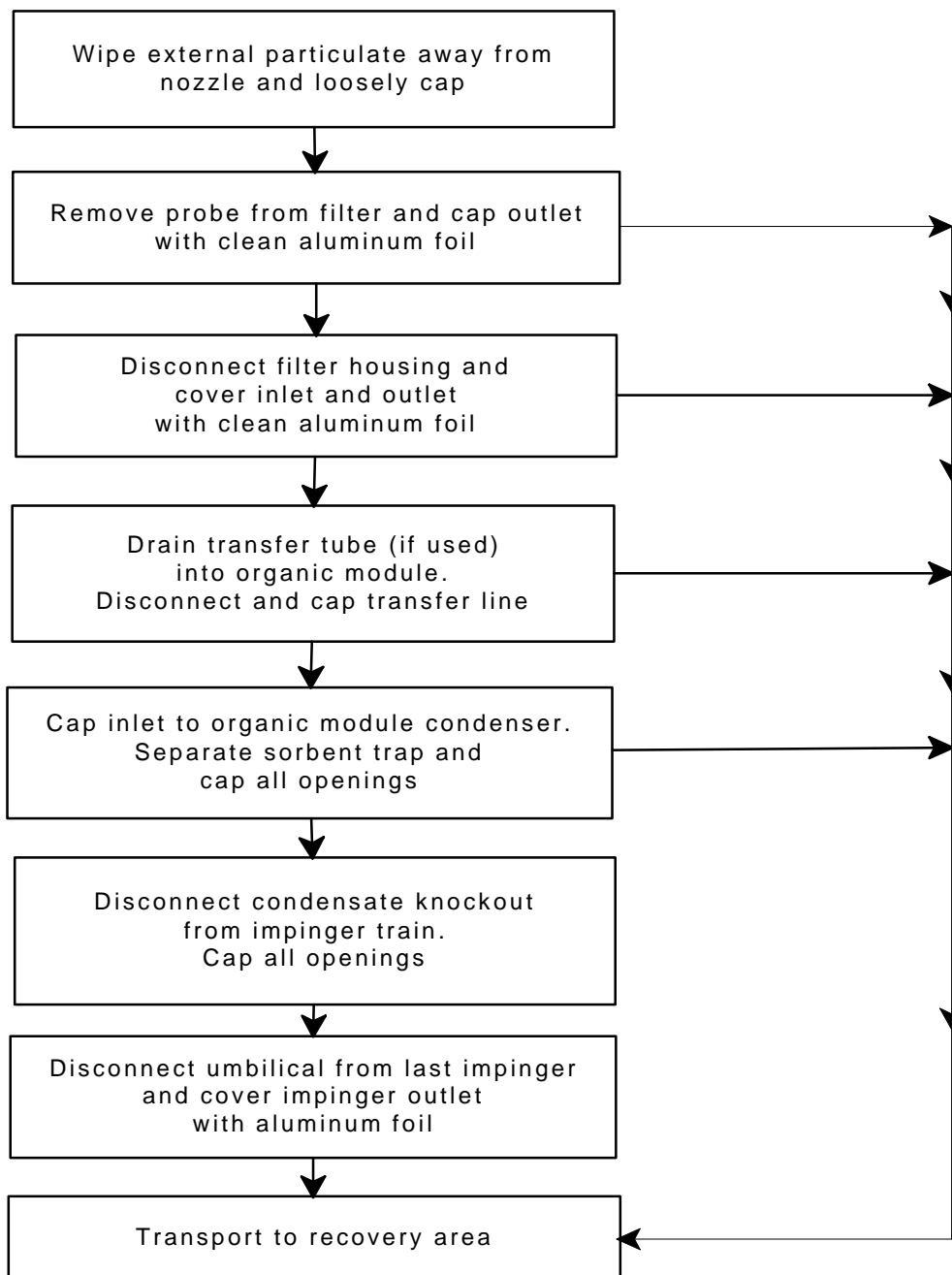


FIGURE A-6 METHOD 0010 SAMPLE TRAIN BREAKDOWN PROCEDURE

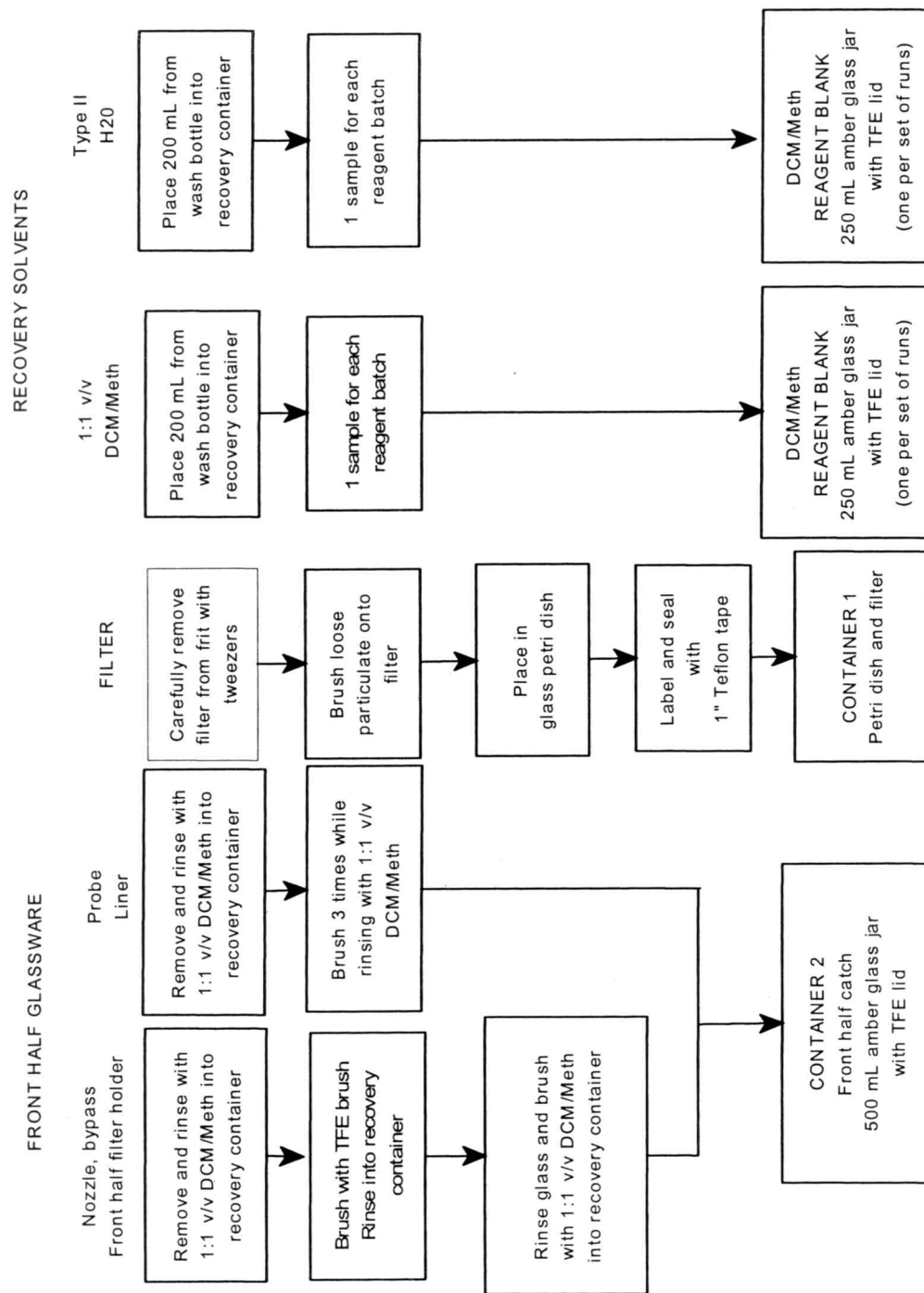


FIGURE A-7

METHOD 0010 SAMPLE RECOVERY PROCEDURE

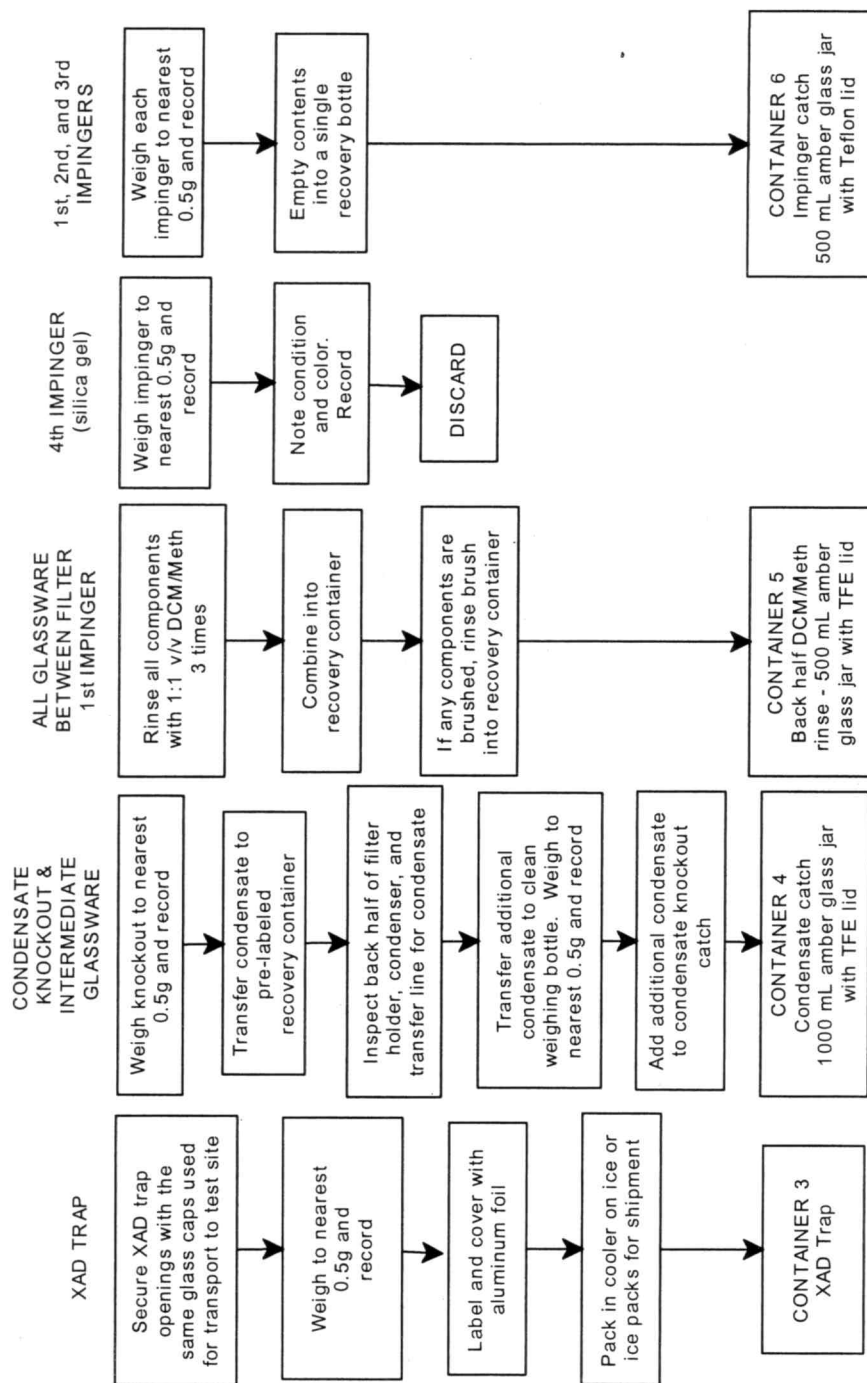


FIGURE A-7 METHOD 0010 SAMPLE RECOVERY PROCEDURE (CONTINUED)

condenser coil, if the sampling platform or sampling port configuration do not allow proper orientation of the sorbent module. The filter in the sampling train will be used to collect organic-laden particulate materials. Gaseous and particulate pollutants, if present, will be withdrawn from the emission source as close to an isokinetic sampling rate as possible. During the melting phase of the project, the high moisture content of the flue gas may prevent isokinetic sampling. The sampling train for Method 23 is illustrated in Figure A-8. An overview of the sampling method is shown in Figure A-9. If the sampling duct does not meet the minimum stack dimension requirements as noted in EPA Method 1, traversing of the duct will not be performed. In that instance, the nozzle of the sampling probe will be positioned in the centroid of the duct for the entire sampling period.

The number of possible interferences with this sampling procedure should be minimal because high resolution gas chromatography coupled with high resolution mass spectrometry will be used during the laboratory analysis. High concentrations of other (nontarget) organic compounds may interfere if the compounds have the same nominal mass as the analytes of interest. Various procedures may be employed to minimize these interferences. Only high purity, residue-free analysis-grade solvents will be used to minimize contaminants introduced from the solvents used in cleanup and recovery. Other possibilities that could result in a positive or negative bias include the following: (1) stability of the extracted compounds in methylene chloride, (2) the formation of water soluble organic salts on the resin in the presence of moisture, and (3) photodegradation of absorbed organic compounds on the resin.

To reduce the chance of sample contamination, all glassware will be thoroughly cleaned before use (see Figure A-10). All glassware and other components (such as Teflon®-coated filter supports, tweezers, Teflon® wash bottles, and probe brushes) will first be washed in hot water with a nonphosphorus laboratory detergent, rinsed with tap water, and then rinsed with deionized

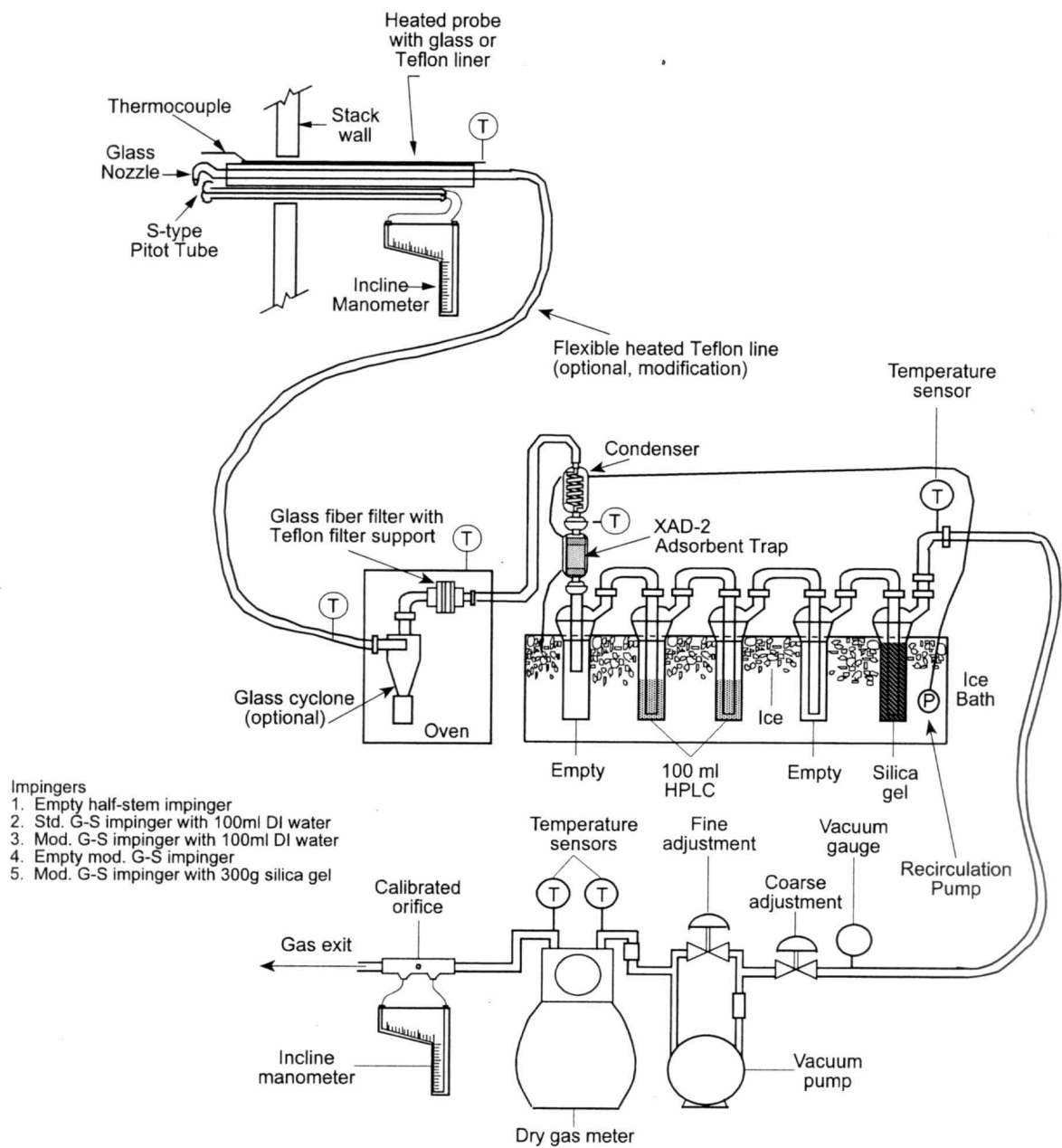


FIGURE A-8 EPA METHOD 23 PCDD/PCDF/PCB SAMPLING TRAIN SCHEMATIC

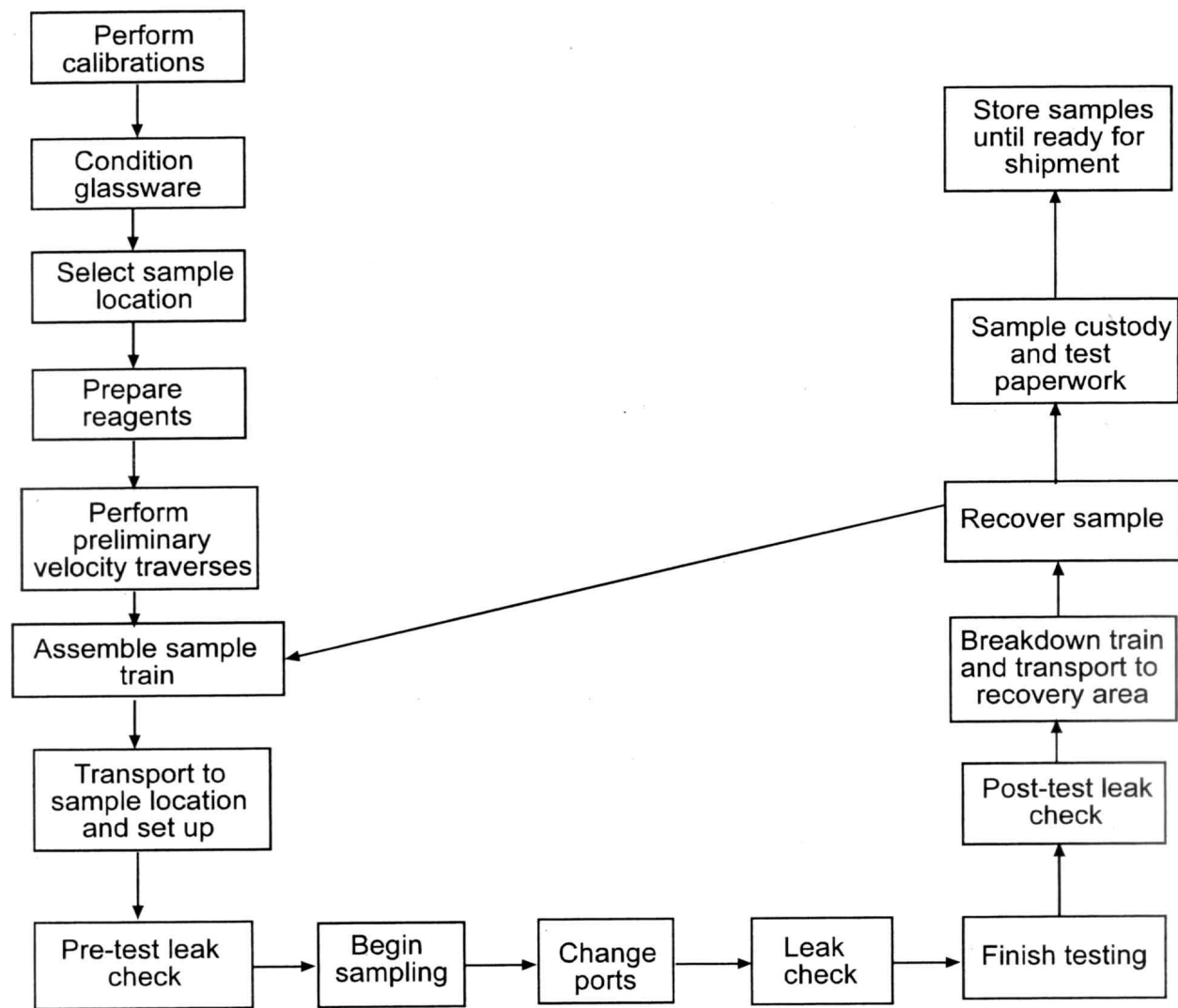


FIGURE A-9

METHOD 23 OVERVIEW

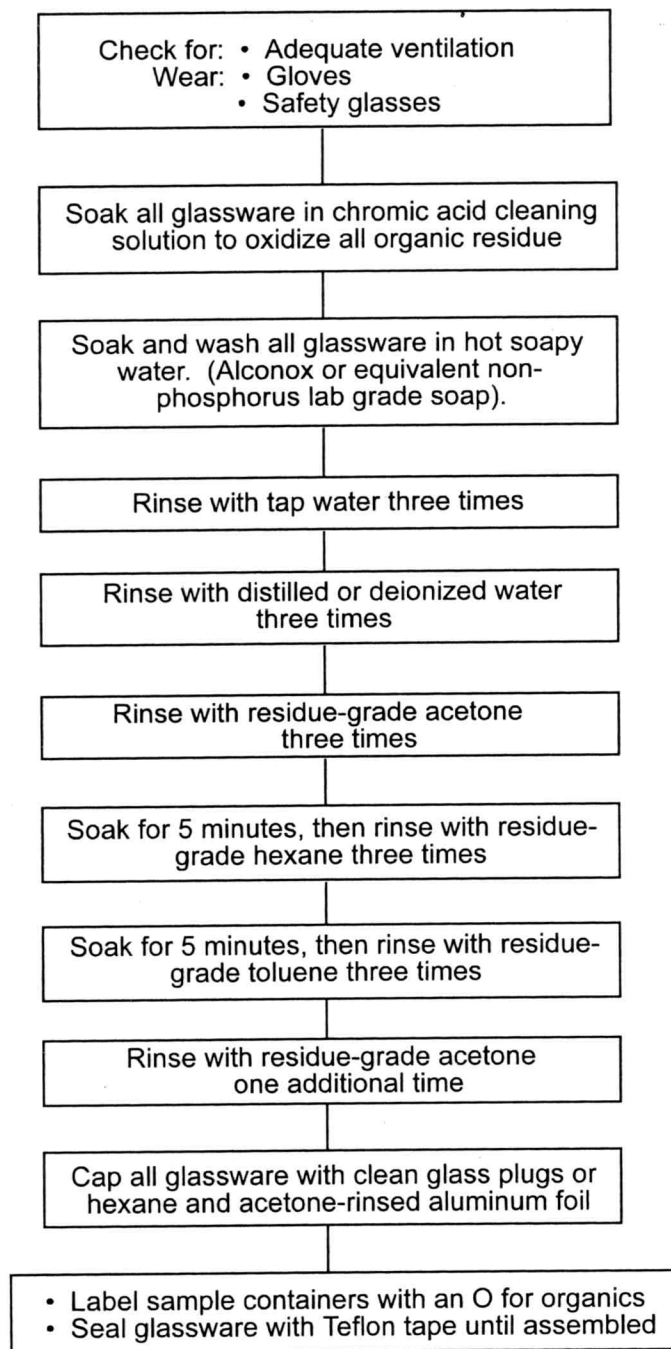


FIGURE A-10 METHOD 23 GLASSWARE PREPARATION PROCEDURE

water. All glassware, excluding volumetric glassware, will then be baked at 450 °C for at least 1 hour. Finally, all components will be rinsed with a series of solvent washes and dried in an organic, solvent-free area.

After cleaning, components will be sealed with glass caps and Teflon[®] tape. All sample recovery containers will be purchased as “certified,”(EPA 1992). Sampling media will be prepared as specified in the methodology by the analytical laboratory. Assembly of the PCDD, PCDF, and PCB sampling train begins in the recovery area. Table A-3 lists the equipment needed to assemble the sampling train. Table A-4 lists the equipment required for recovery of the sampling train. During the assembly of the train, a visual inspection of each of the sampling train components and equipment will be performed. Glassware will be checked for cracks and chips, and all connections will be cleaned to ensure good seals. Fittings and ferrules will be inspected for wear, and tubing are inspected for wear and flexibility. All damaged or defective components of the sampling train will be replaced. All openings of the train will be covered with aluminum foil or Teflon[®] tape during the assembly process. No silicone grease will be used on any ground glass joints prior to the organic resin module. The probe will be constructed of a single, heated, quartz glass tube, bent at the nozzle. The temperature of the probe will be monitored by a thermocouple placed under the probe heater in the back half of the probe. The nozzle is tapered to meet sharp-edged nozzle requirements. During the melting phase of the program, if the temperature of the flue is greater than 900 °F, an air-cooled probe may be used, where the outer sheath of the probe is jacketed with a flow of compressed air. This allows the interior probe liner to be maintained at a constant temperature without absorbing the radiant heat from the flue. The use of the air-cooled probe is not anticipated.

The temperature of the optional heated transfer line will be monitored by a thermocouple inserted between the Teflon[®] tube and the heating jacket near the probe-end of the line (if the heated

TABLE A-3 SAMPLING TRAIN COMPONENT CHECKLIST FOR METHOD 23

Probe nozzle	- Quartz or borosilicate glass
Probe liner	- Borosilicate glass or quartz tubing
Probe sheath and heater	- Stainless-steel (316) probe sheath capable of maintaining the exit or the probe at $120\text{ }^{\circ}\text{C} \pm 12\text{ }^{\circ}\text{C}$
Pitot tube	- Type S (as per EPA Method 2)
Differential pressure gauge	- Inclined manometer (as per EPA Method 2)
Glass Cyclone	- Borosilicate glass, used for high particulate sample stream (optional)
Filter holder	- Borosilicate glass with a Teflon [®] frit filter support and gasket
Filter	- Precleaned and prespiked glass fiber filter with no organic binder
Filter heating system	- Capable of maintaining the filter holder at $120\text{ }^{\circ}\text{C} \pm 12\text{ }^{\circ}\text{C}$
Organic sampling module	- Unit consists of a gas-conditioning section, a spiked sorbent trap, and a condensate knockout trap sized to remove moisture from the sample
Water circulating bath	- Bath and pump capable of circulating water to the condensor and sorbent trap water jackets
Crushed Ice	- 10 to 50 Pounds depending on the ambient temperature
Impinger train	- Three 500-ml impingers, connected in series with leak-free, ground-glass joints
Metering system	- Vacuum gauge, leak-free pump, thermometers, dry gas meter, and related equipment, as shown in Figure 4-1
Barometer	- Aneroid or other barometer capable of measuring to within 2.5 mm Hg
Gas density equipment	- Temperature sensor and pressure gauge, and gas analyzer
Calibration and field record	- A permanently bound laboratory notebook

TABLE A-4 SAMPLE RECOVERY CHECKLIST FOR METHOD 23

Probe liner brush	- Nylon or Teflon [®] bristle brush with stainless-steel wire or Teflon [®] handle, Teflon [®] extensions at least as long as the probe may be used
Wash bottles	- Teflon [®] , 500 ml
Sample storage containers	- Chemically resistant, borosilicate amber and clear glass with screw-cap liners made of Teflon [®] (1,000 ml, 500 ml, 250 ml)
Petri dishes	- Glass, sealed with Teflon [®] tape for transportation of samples
Graduated cylinder/balance	- To measure condensed water to nearest 0.5 ml or 0.5 g
Plastic storage containers	- Screw-cap polypropylene or polyethylene for silica gel
Funnel and rubber policeman	- To aid in the transfer of silica gel
Funnels	- Glass, to aid in sample recovery
Filters	- Spiked glass fiber, without organic binder, 99.95 percent efficient
Teflon [®] tweezers	- For filter recovery
Crushed ice	- For cooling train during the run and for transport of samples
Stopcock grease	- Solvent-insoluble, heat-stable silicone grease
Glass wool	- Used to plug the unfritted end of the sorbent module
Adsorbent resin	- Porous polymeric resin (XAD-2 [®] or equivalent), cleaned prior to use
Silica gel	- Indicating type, 6 to 16 mesh
Impinger solutions	- Distilled organic-free water (Type II) 1N NaOH
Methylene chloride	- Distilled-in-glass or residue analyzed grade
Methanol	- Distilled-in-glass or residue analyzed grade
Water	- Distilled organic-free (Type II)

transfer line is used). The filter assembly will be installed in a temperature-controlled oven to maintain a temperature of $248^{\circ}\text{F} \pm 25^{\circ}\text{F}$. The filter assembly is constructed of borosilicate glass, and the filter support is constructed of Teflon[®]. A water-cooled condenser is located downstream of the heated filter oven. The condenser is followed by a water-cooled sorbent module containing about 50 g of precleaned XAD-2 resin. The analytical laboratory will prepare and load the XAD-2 resin into the absorbent cartridges according to EPA Method 23 by the laboratory. The laboratory will supply filters of the appropriate size to use in the sampling train. The filters will be cleaned to remove any organic matter. The filters must exhibit at least 99.95 percent RE on $0.3\text{ }\mu\text{m}$ dioctyl phthalate smoke particles (typically vendor documentation of this is sufficient). Each XAD-2 resin cartridge and filter will be spiked with isotopically labeled PCDD, PCDF or PCB congeners reference standards by the lab (see Section 5.0) to determine if any loss of surrogates occurred during sample handling, shipment, or analysis. During sampling, the XAD-2 cartridge inlet temperature will be monitored to ensure that the temperature of the flue gas sample entering the module is maintained below 20°C to ensure sufficient capture of organics.

The XAD-2 resin module is located above an empty impinger. The impinger is used to collect any condensate in the flue gas sample. The first impinger is modified without a stem so the sample gas does not bubble through the collected condensate. An additional empty impinger may be added if the stack moisture conditions yield a large volume of condensate. The second impinger in the sampling train is of Greenberg-Smith (GS) design. The third and fourth impingers will be of modified GS design. The second impinger will contain 100 ml of organic-free Type II water. The third impinger will be charged with 1 N sodium hydroxide to scrub any hydrogen chloride and sulfur dioxide from the sample stream prior to the pump. The fourth will contain a known weight of silica gel. Impinger weights and contents will be recorded on an impinger weigh sheet, which stays with the impinger set during the test. All connections within the train will be glass or Teflon[®]. The impingers are followed by a meter box containing a pump, dry gas meter, and a calibrated orifice meter. Assembly procedures are shown in

Figure A-11.

Following completion of the organics train pre-test checklist, the operator informs the crew chief and awaits instructions to position the probe at the first traverse point (if traversing is required) and begin sampling. Stack gas velocity is measured at each traverse sampling point with an s-type pitot probe. If the duct diameter is less than 12 inches, the duct will not be traversed, and the probe tip will be located in the centroid of the duct. A hand calculator is used to calculate the orifice meter reading corresponding to isokinetic flow. All field data will be recorded using black ballpoint pens.

During the run, the filter will be maintained at a minimum of $248^{\circ}\text{F} \pm 25^{\circ}\text{F}$ ($120^{\circ}\text{C} \pm 14^{\circ}\text{C}$), and the gas entering the sorbent trap will be maintained at a maximum of 60°F (20°C). The sampling ports will be inspected and cleaned prior to any sampling activities. The sampling train will be leak checked at the sampling site, after the train has reached sampling temperature and prior to any sampling activities. The sampling pump will be started with the fine adjustment completely open and the course adjustments completely closed. The coarse adjustment will be opened slowly while the fine adjustment is being closed until a vacuum of 15 mm Hg is reached. A leak rate of no more than 0.00057 cubic meters per minute (m^3/min) or 0.02 cubic feet per minute (cfm) or 4 percent of the sampling rate, whichever is less. Leak check procedures are shown in Figure A-12.

When sampling has been completed, as soon as the probe is withdrawn from the sampling port, the nozzle will be covered with pre-cleaned aluminum foil, and the probe will be disconnected from the filter holder. The train components will be allowed to cool until they can be handled safely. All external particulate matter will then be wiped from the probe, then both ends will be

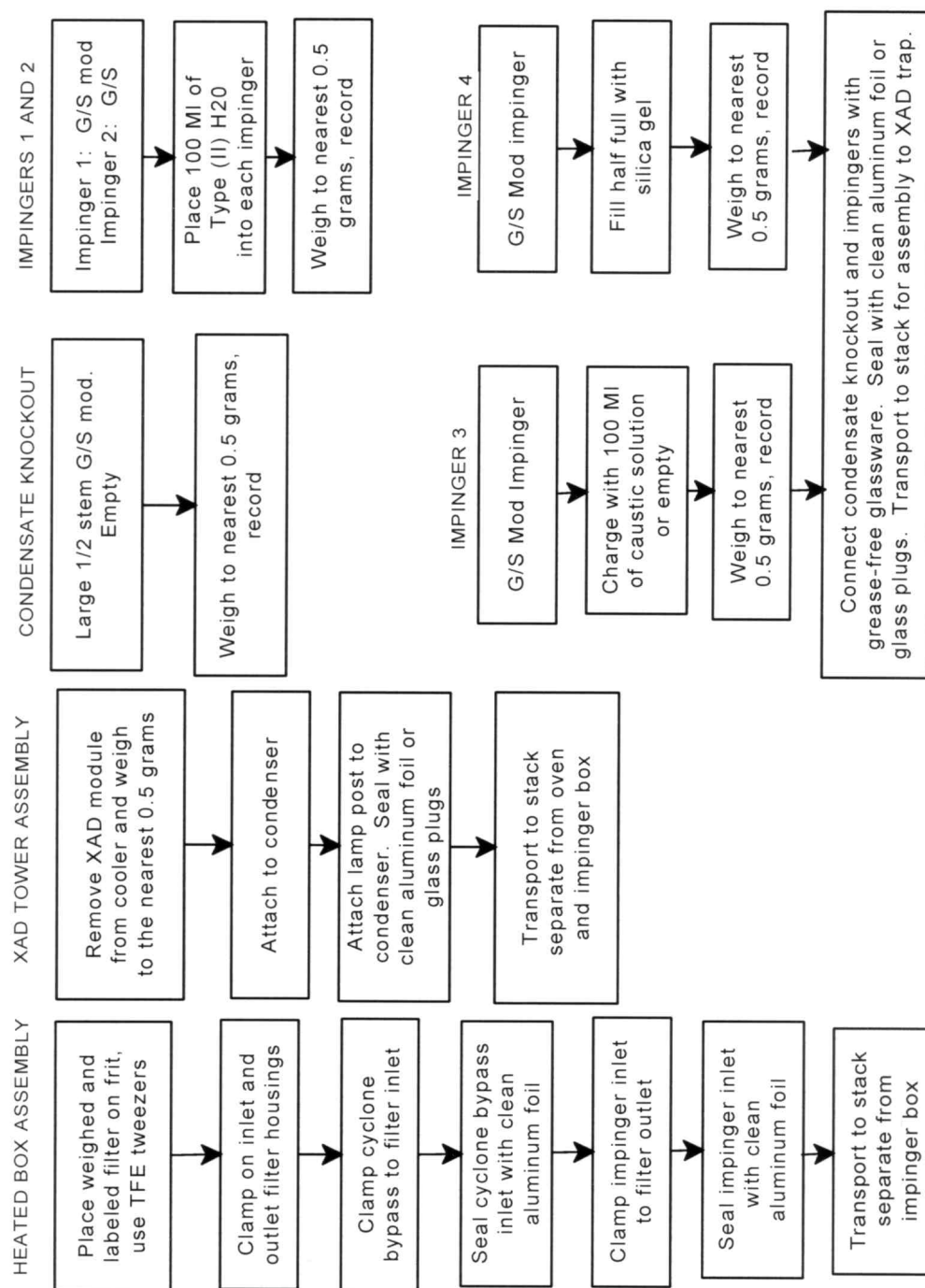


FIGURE A-11 METHOD 23 - SAMPLE TRAIN ASSEMBLY PROCEDURE

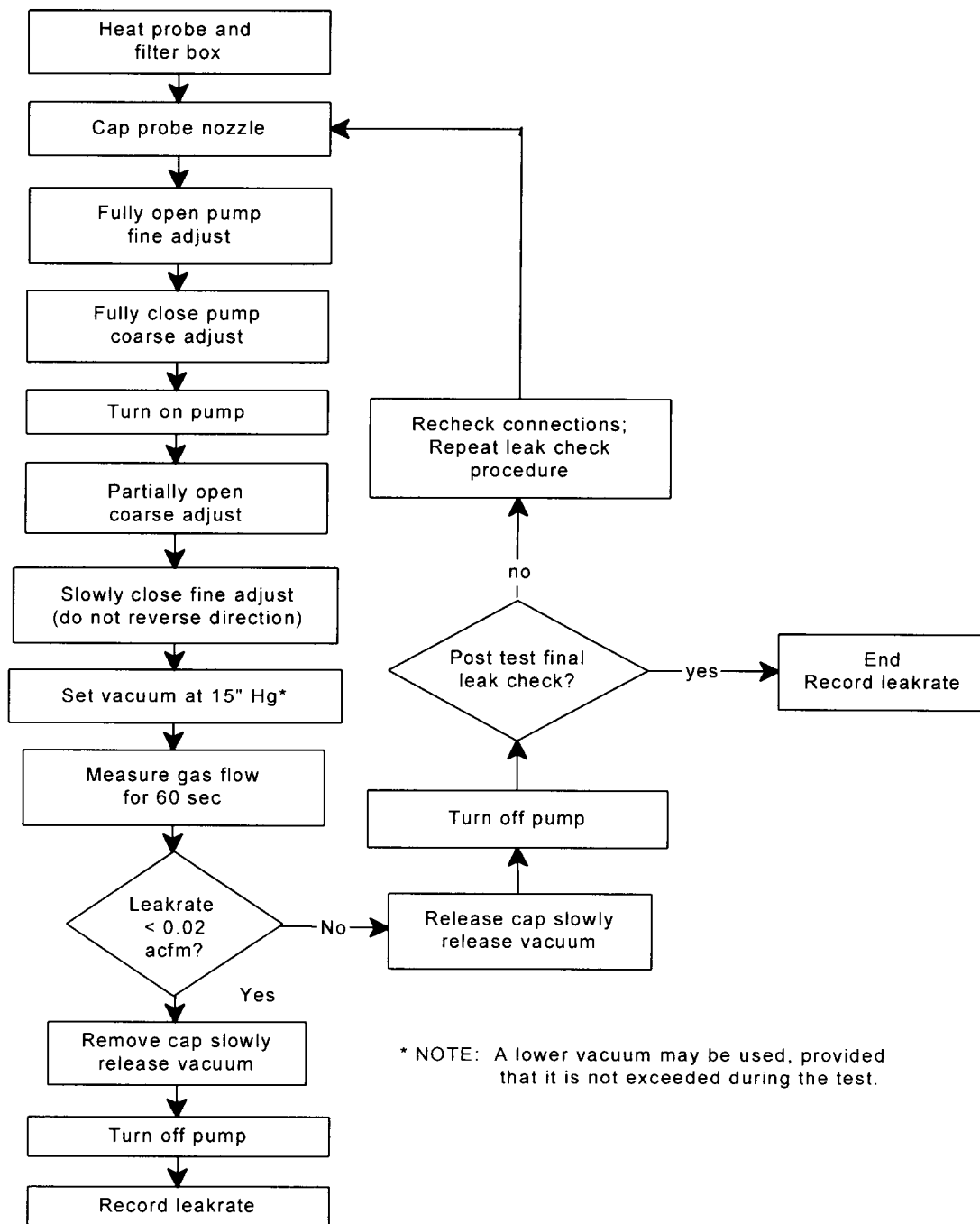


FIGURE A-12

METHOD 23 LEAK CHECK PROCEDURE

sealed with aluminum foil. Both openings of the filter holder, the transfer line (if used), condenser, sorbent trap, and the impinger train will be sealed. The sample train breakdown scheme is illustrated in Figure A-13. All components of the sample train will be delivered to the recovery area. The probe and flexible line will then be rinsed with the specified solvents into a socket flask attached directly to the end of the probe or line. This approach reduces the possibility of sample loss during recovery. The recovery containers for the rinseate liquids will be contaminant-free, 1,000- or 500-ml amber glass bottles with Teflon[®]-lined lids. Glass petri dishes will be used for filter storage. XAD-2 resin modules will be capped with prewashed glass plugs. Following recovery, the sample train will be reassembled with a fresh XAD-2 cartridge, and all openings will be sealed with prewashed glass plugs. Figure A-14 shows the recovery scheme to be used for this test program. Once recovered, all sorbent traps and rinseates will be either stored on ice or refrigerated at 4°C until transported to the laboratory. The filters will be stored separately at ambient temperature until transported to the laboratory. The filters and XAD-2 will be wrapped in aluminum foil to minimize photodegradation.

A.5 EPA Method 0031 -- Volatile Organic Compounds

VOCs in gaseous emissions from the stack of the melter will be collected using EPA Method 0031. VOCs will just be collected during the vitrification phase of the program. In the sampling method for VOCs (SM-VOC), a 20-liter sample of effluent gas is withdrawn from a combustion source at a flow rate of 1 liter per minute (L/min) over a period of 20 minutes. Alternately, a sampling rate of 0.5 L/min over a period of 40 minutes may be used (SLO-SMVOC) when the boiling points of compounds of interest are less than 0 °C. For this sampling program, the SLO- SMVOC train will be used because the expected flow rate from the melter is expected to be low. A schematic of the sampling train is shown in Figure A-15. An overview of the sampling procedure is shown in Figure A-16.

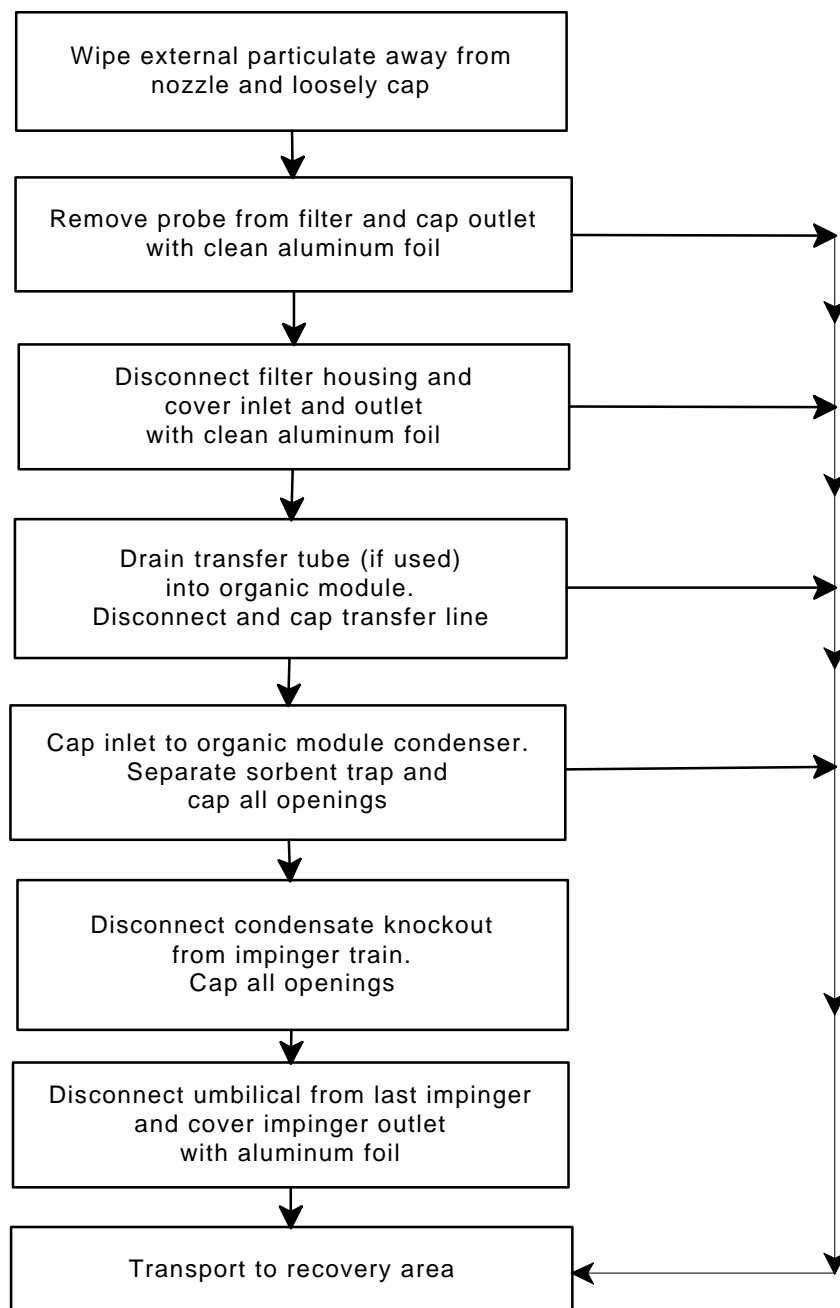
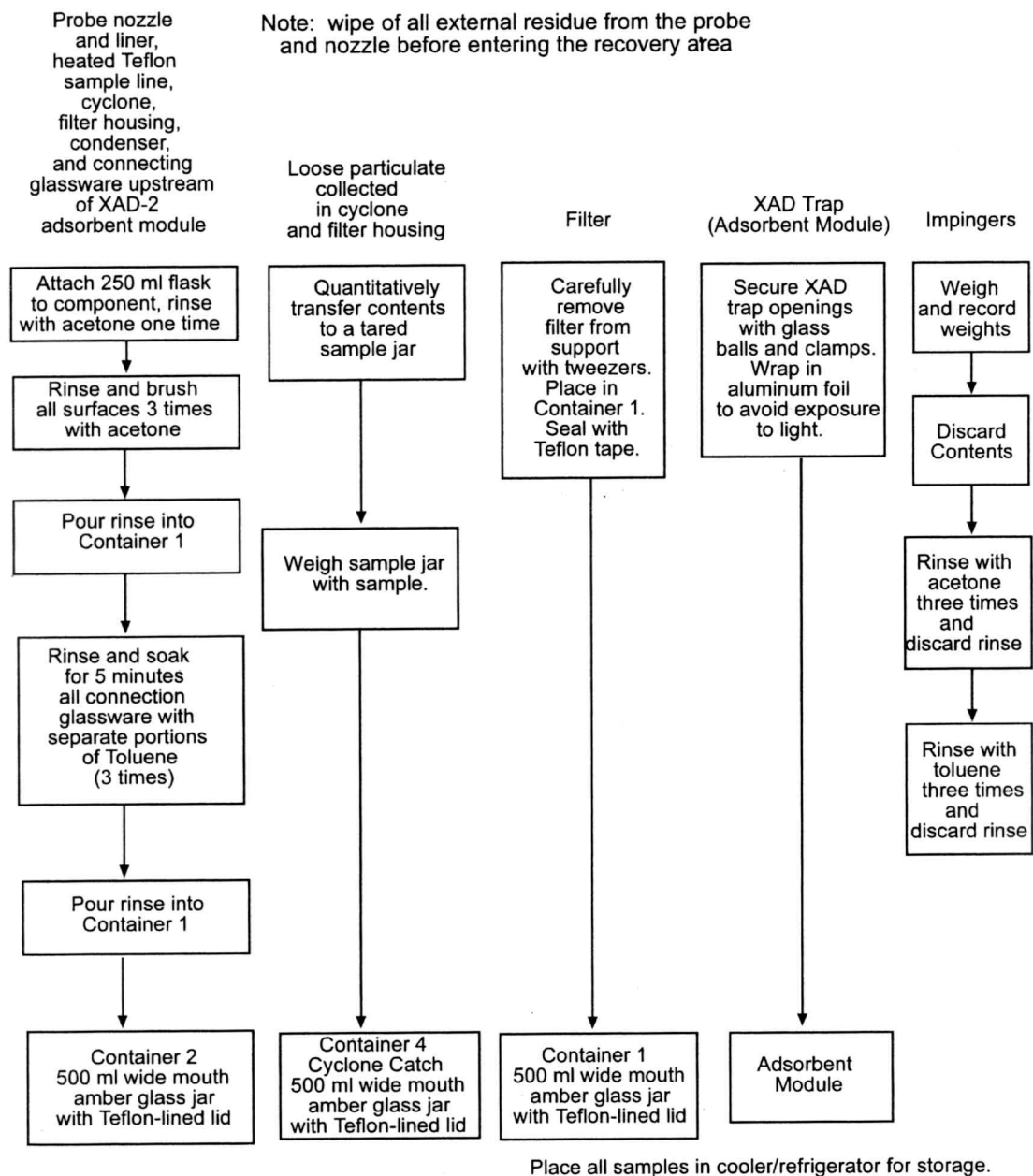


FIGURE A-13 METHOD 23 SAMPLING TRAIN BREAKDOWN SCHEME



EPA Method 23 sample train field recovery scheme, modified to eliminate methylene chloride rinse, combine toluene and acetone rinses, and separate recovery of loose particulate matter.

FIGURE A-14

METHOD 23 SAMPLE RECOVERY PROCEDURE

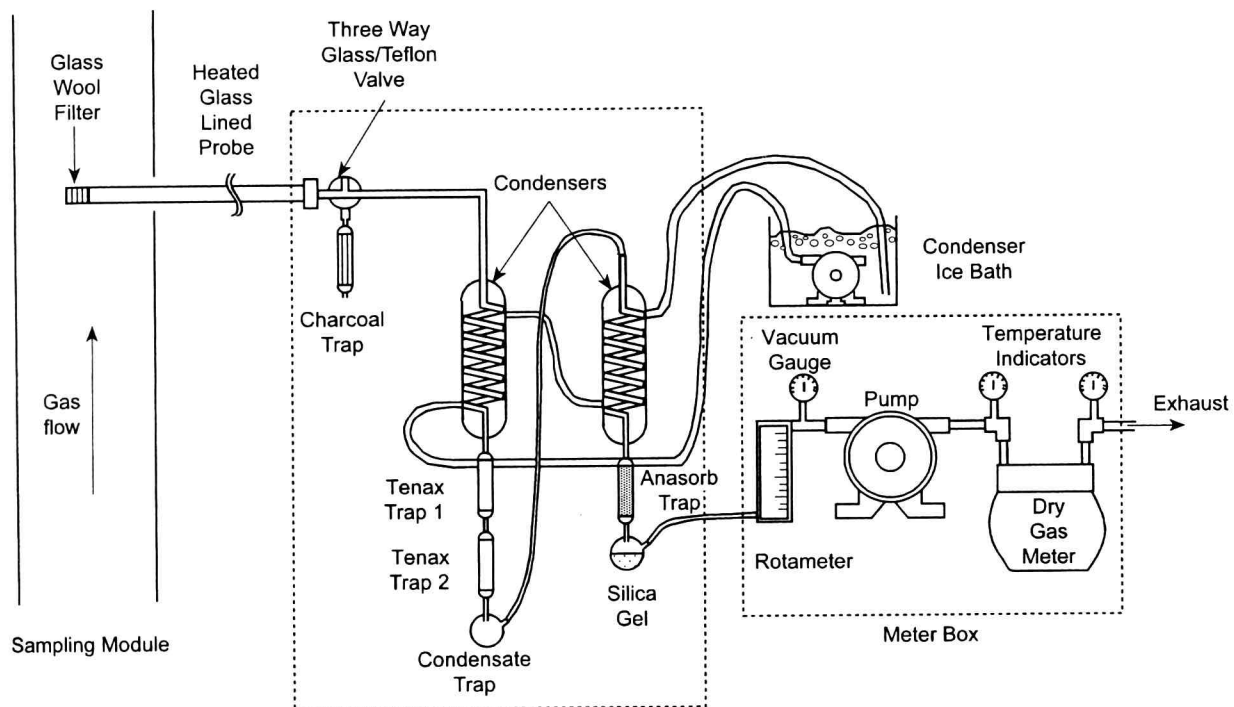


FIGURE A-15 METHOD 0031 VOLATILE SAMPLING TRAIN SCHEMATIC

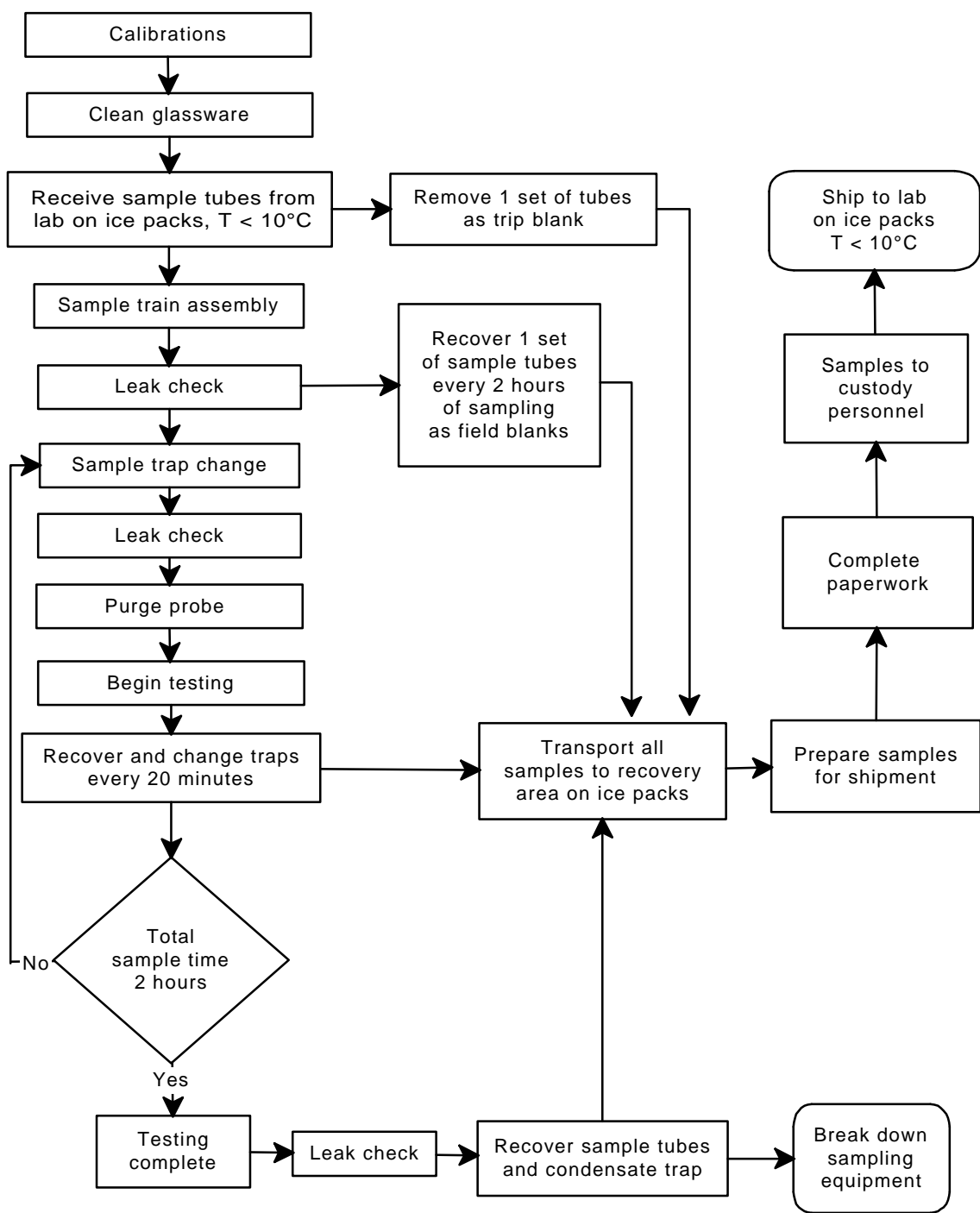


FIGURE A-16

METHOD 0031 OVERVIEW

EPA Method 0031 is applicable for most VOCs that have a boiling point between -15 °C and 121 °C. It is not applicable for polar, water-soluble, and reactive organic compounds. This method is also not applicable when the concentration of a given organic compound exceeds 1.5 ppm. Saturation of the collection media and breakthrough can occur, invalidating the final results. The sensitivity of this method is driven by the level of interferences in the sample matrix and the presence of detectable levels of a given VOCs. Generally, laboratories have been able to demonstrate detection limits of 0.1 to 100 micrograms per cubic meter (: g/m³) using this method.

This gas sampling method uses a sampling probe, three adsorbent modules, and a low-flow meter box to extract the sample from the source. The sample is extracted from the sampling port through a heated, glass-lined sample probe with a filter at the nozzle end. After exiting the probe, the gases are cooled to 20 °C using a water-cooled condenser, and the organics are collected on a series of three resin traps: two Tenax[®]-GC and one Anasorb[®]-747. Liquid condensate is collected in an impinger placed after the two Tenax[®]-GC traps and before the Anasorb[®]-747 trap. The first and second traps each contain 1.6 g of Tenax[®]-GC each, and the third trap contains 5.0 g of Anasorb[®]-747. Tables A-5 and A-6 list the components and reagents required for assembly and recovery of the sampling train used for this method.

Possible interferences may be encountered in the analytical methodology and may arise primarily from background contamination of sorbent traps prior to or after sampling. Other interferences may arise from (1) exposure of the sorbent materials to solvent vapors prior to assembly and (2) exposure to significant concentrations of VOCs in the ambient air at a stationary source site. To minimize the low-level contamination of train components with VOCs care will be taken to avoid contact of all interior surfaces of the train components with synthetic organic materials, such as organic solvents and lubricating and sealing greases. Train components will be carefully

TABLE A-SAMPLING TRAIN COMPONENT CHECKLIST FOR METHOD 0031

Probe liner	- Borosilicate glass or quartz with a plug of clean quartz wool inserted in the tip
Probe sheath*	- Stainless-steel (316) maintained at $130\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$ ($266\text{ }^{\circ}\text{F} \pm 9\text{ }^{\circ}\text{F}$).
Isolation valve	- Grease-less stopcock (0.25-inch outside diameter) with a glass bore and sliding Teflon [®] plug with Teflon [®] washers
Condensers	- Two Ace Glass 5979-14 or equivalent with the capacity to cool the gas stream to $20\text{ }^{\circ}\text{C}$ or less prior to passage through the first sorbent tube
Metering system	- Vacuum gauge, pump, thermometers, dry gas meter (2 percent accuracy, minimum resolution of 0.01 L), calibrated rotameter, needle valves, and a temperature readout
Sorbent tubes	- Glass tubes with dimensions of 10 centimeters (cm) x 1.6 cm inside diameter. Two tubes packed with 1.6 g of Tenax-GC [®] and one tube packed with 5.0 g of Anasorb [®] -747.
Condensate trap	- Borosilicate glass
Sample transfer lines	- Heat-traced Teflon [®] or glass maintained at $130^{\circ}\text{C} \pm 5^{\circ}\text{C}$, less than 1.52 m (5 ft.) long; Between probe and first condenser
Sample transfer lines	- Teflon [®] with connecting fittings that will form a leak-free seal; between condensate trap and second condenser
Calibration/field record-	A permanently bound laboratory notebook
Silica gel	- Indicating type, 6 to 16 mesh; silica gel should not be reused.
Glass wool	- Glass wool shall be Soxhlet extracted for 8 to 16 hours using methanol, then oven dried at $110\text{ }^{\circ}\text{C}$ before use
Quartz wool	- Used for high temperature applications, greater than $150\text{ }^{\circ}\text{C}$.
Teflon [®] wash bottle -	For "Volatile-Free" water

**No stainless steel can contact any sampling surface*

TABLE A-6 SAMPLE RECOVERY CHECKLIST FOR METHOD 0031

Cold packs/Ice	- Cold packs or ice should be used to maintain tubes at less than 10 °C; tubes should not be exposed to water when ice melts.
VOC Vials	- 40-ml glass vials with Teflon [®] -lined caps for condensate recovery
Teflon [®] squeeze bottles -	Prior to use, bottles should be washed with laboratory detergent, rinsed with hot tap water, then distilled water, then rinsed with clean purged water. Use new bottles, not one that has had a solvent in it in the past.
2,6-Diphenyl-D-phenylene	- New Tenax [®] -GC, 35/60 mesh is Soxhlet extracted for 24 hours with oxide polymer Methanol and dried for 6 hours in a vacuum oven at 50 °C before use. Thermal conditioning should be done prior to blanking. Reuse of sorbents is not recommended.
Anasorb [®] -747	- New Anasorb [®] -747 is used as it is received from the manufacturer without preparation other than thermal conditioning pending a QC check. Anasorb [®] -747 must not be reused or extracted with organic solvents prior to use as a sorbent in the SMVOC
Water	- “Volatile-free” Type I water should be used. Water used to recover the condensate in the SMVOC should be provided by the analytical laboratory and from which all volatile organic compounds have been purged. Purged water is prepared by either purging the water with nitrogen for several hours or drawing the water through and adsorption cartridge. The purged water is then stored in a bottle with a Teflon [®] -lined screw cap and added to a Teflon [®] wash bottle before use.

cleaned and conditioned according to the procedures described in this protocol (see Figure 4-17).

Figure A-18 shows the configuration for the sorbent tubes (Tenax[®]-GC and Anasorb[®]-747) used for sample collection. The use of blanks is essential to assess the extent of any background contamination.

After assembling the train (see Figure A-19) and leak checking (see Figure A-20), but before sample collection, the probe will be purged with stack gas by pulling a sample through the isolation valve using a secondary vacuum pump. Sample collection is accomplished by opening the valve at the inlet of the first condenser and turning on the pump. Three sorbent tube sets will be collected over a 2-hour sampling period. A sampling rate of 0.5 L/min for 40 minutes will be used for each run. Under no circumstances will the sampling volume exceed 20 L for a given set of tubes. During sampling, the sorbent tube caps will be stored in clean, organic-free glass containers with Teflon[®] lined screw caps. Following each 40-minute sampling period, the Tenax and Anasorb sorbent tubes will be recovered as a set and will be replaced with the next set of sorbent tubes. If high moisture conditions exists and the volume of condensate from a 20-L gas sample will exceed 40 ml, the condensate trap will be removed with each set of sorbent tubes. The contents of all condensate traps will be combined during sample recovery. After replacing the sorbent tubes with a new set, the system is again leaked checked at the highest vacuum experienced during the sample run to minimize vacuum volatile deposition off the sorbent traps.

Recovery of this sampling train involves rinsing the condenser with “volatile-free” water prior to the removal of the sorbent cartridges. Condensate and the condensate trap rinse will then be collected into a preserved 40-ml VOC vials with Teflon[®] lined caps. If any headspace exists in the 40-ml vial, it must be eliminated by adding enough “volatile-free” water to overfill the vial. The samples will be stored at $4\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$ in a cooler until receipt at the laboratory. Figure A-21 lists the recovery procedure.

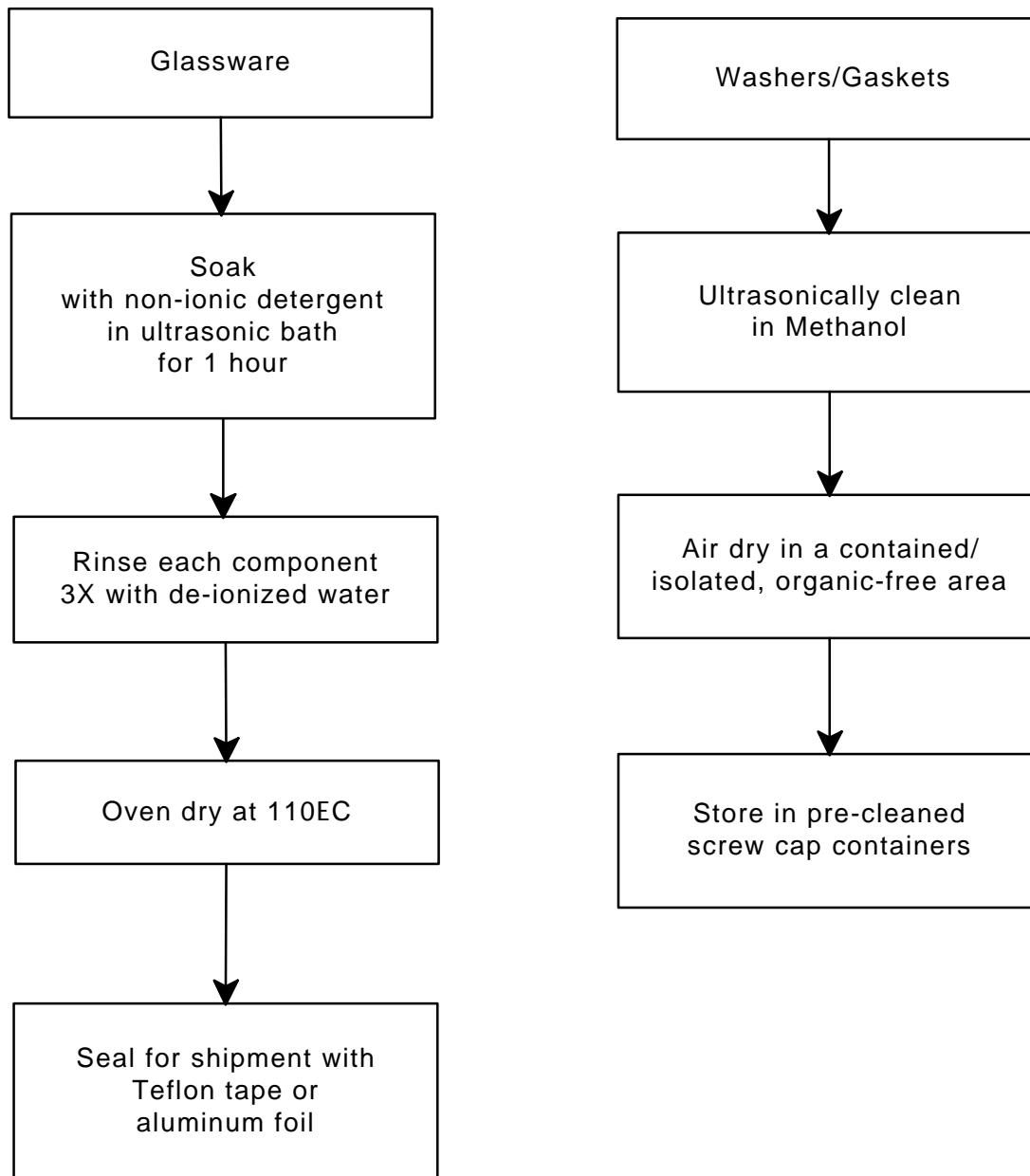


FIGURE A-17 METHOD 0031 GLASSWARE PREPARATION PROCEDURE

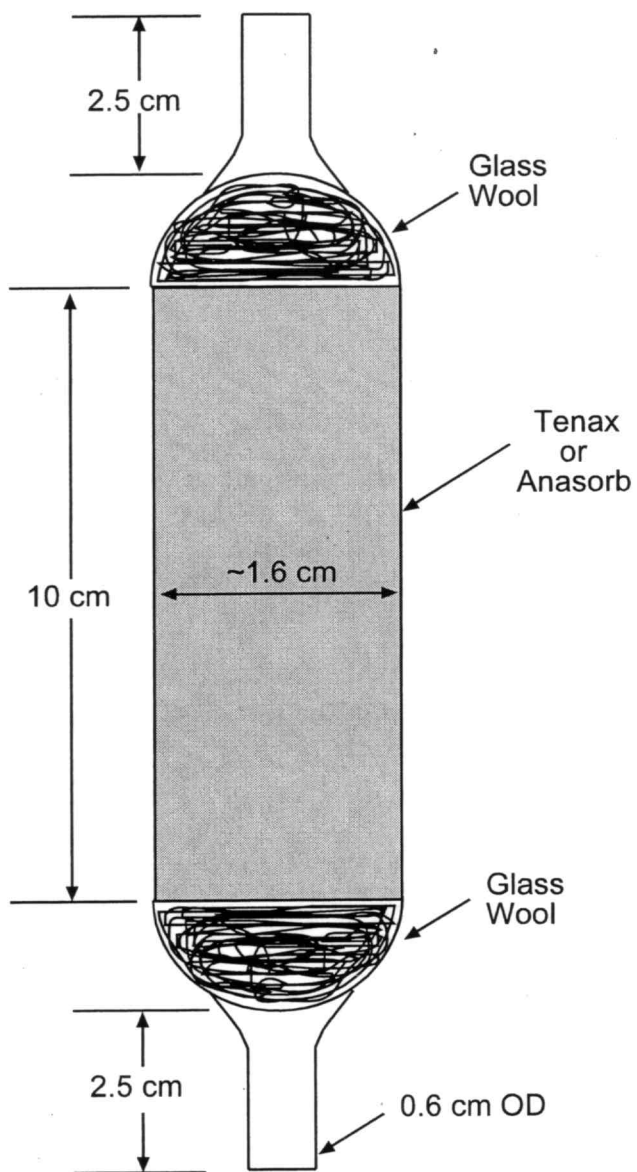


FIGURE A-18

METHOD 0031 SORBENT TUBE CONFIGURATION

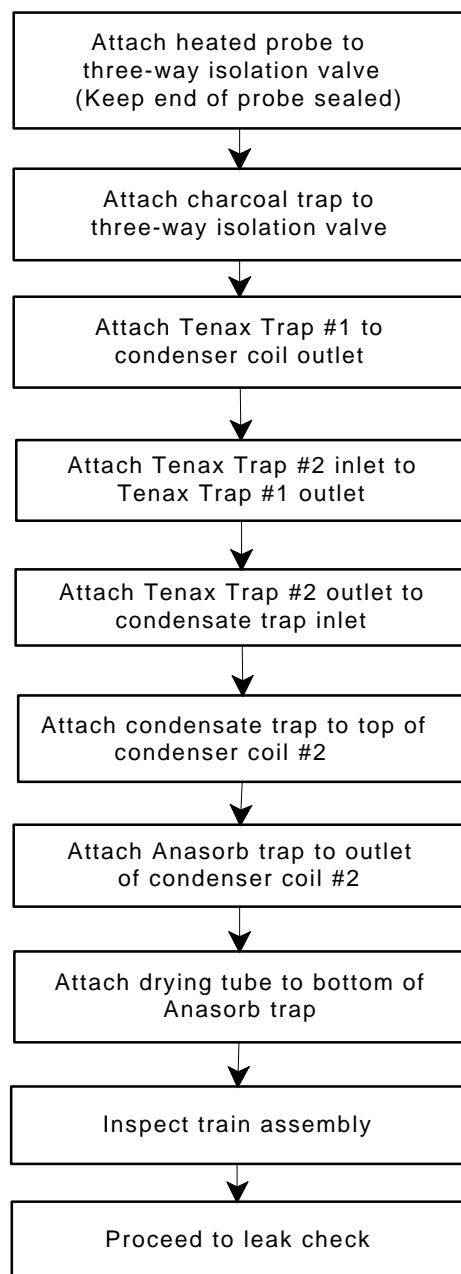


FIGURE A-19 METHOD 0031 SAMPLING TRAIN ASSEMBLY PROCEDURE

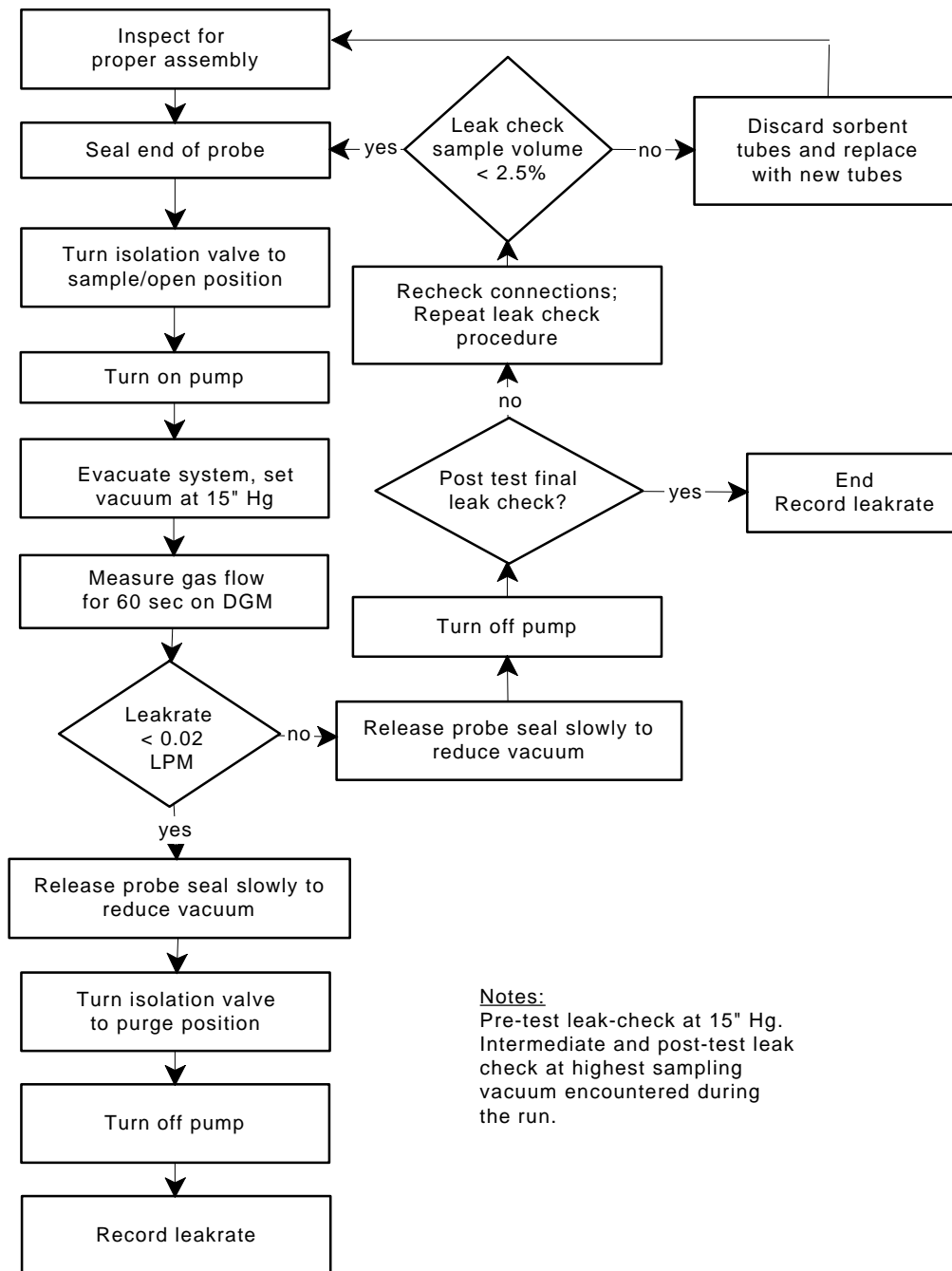


FIGURE A-20

METHOD 0031 LEAK CHECK PROCEDURE

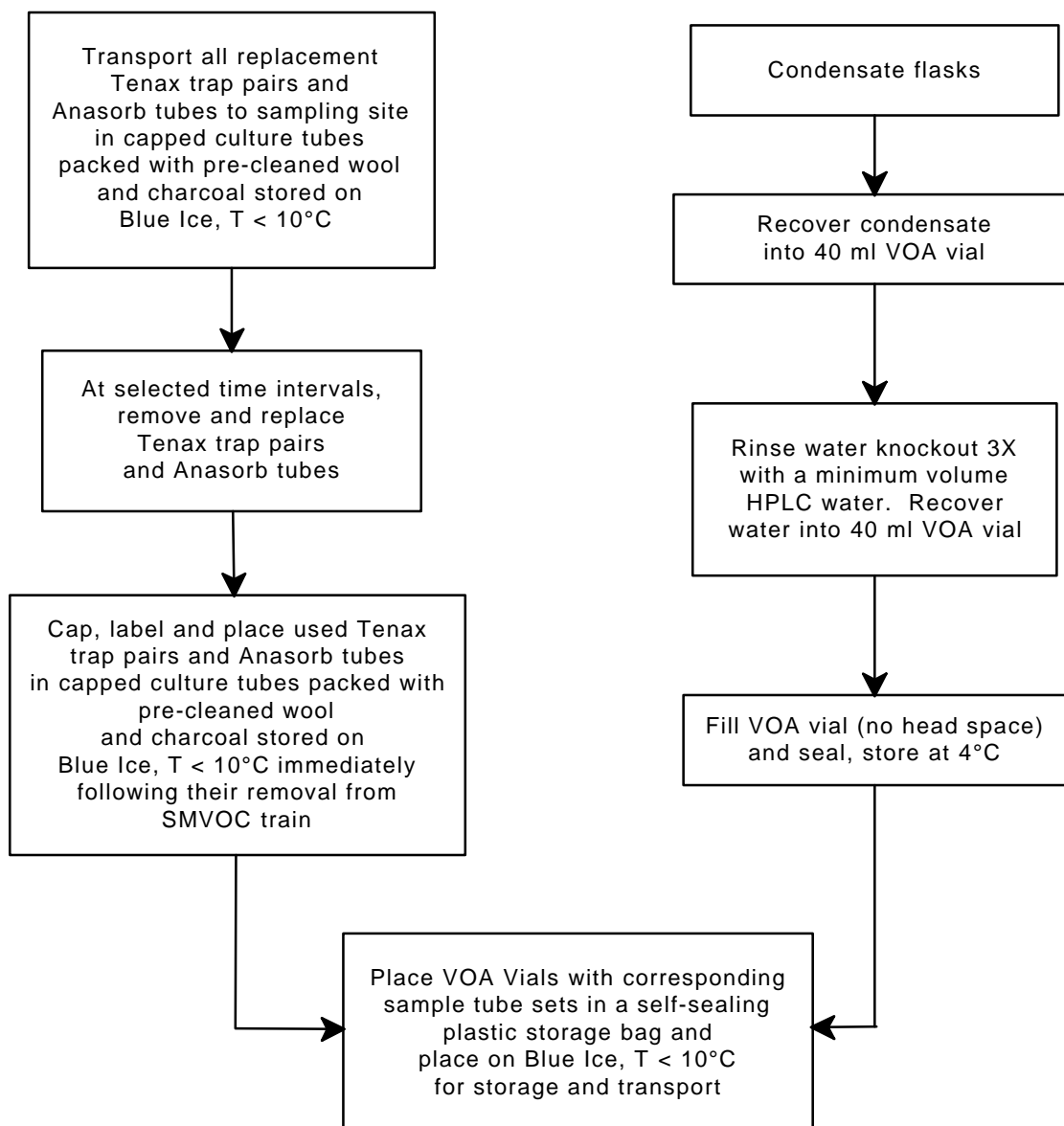


FIGURE A-21

METHOD 0031 SAMPLE RECOVERY PROCEDURE

A.6 EPA Method 0060 - Metals

Trace metals in the gaseous emissions from the remediation system will be collected at the G1 (well field) and G2 (system stack) locations using EPA Method 0060. This sampling method is based on EPA Method 29. The collected samples will be transported to the analytical laboratory for preparation and analysis. It is expected that only volatile metal species (Mercury, Arsenic, Lead, Selenium, Thallium, and Antimony) will be detected in the process emissions. Gaseous and particulate pollutants will be withdrawn from the emission source at an isokinetic rate and collected in the sampling train illustrated in Figure A-22. If the minimum stack dimension requirements, as specified in Method 1, are not met, the stack will not be traversed, but the probe nozzle will be positioned in the centroid of the duct for sampling. Particulate emissions are not expected to be present in the emissions from the system. If particulate matter is present, it will be collected in the probe and on the heated filter. Gaseous emissions are collected in a series of chilled impingers. An overview of the sampling procedure is illustrated in Figure A-23.

Assembly of the metals sampling train begins in the recovery area. Tables A-7 and A-8 list the equipment needed to assemble the sampling train and recover the sampling train. During the assembly of the train, a visual inspection of each of the sampling train components and equipment is performed. Glassware will be checked for cracks and chips, all connections will be cleaned to ensure good seals. Fittings and components will be sealed with glass caps and Teflon[®] tape. All sample recovery containers will be purchased as “Certified”, indicating the containers have been cleaned to U.S. EPA Contract Laboratory Program analyte specifications.

Following assembly of the multi-metals train, the train is transported to the sampling location. The sampling port is cleaned to prevent contamination from particulate matter present in the port cover. The operator then informs the crew chief and awaits instructions to position the probe at

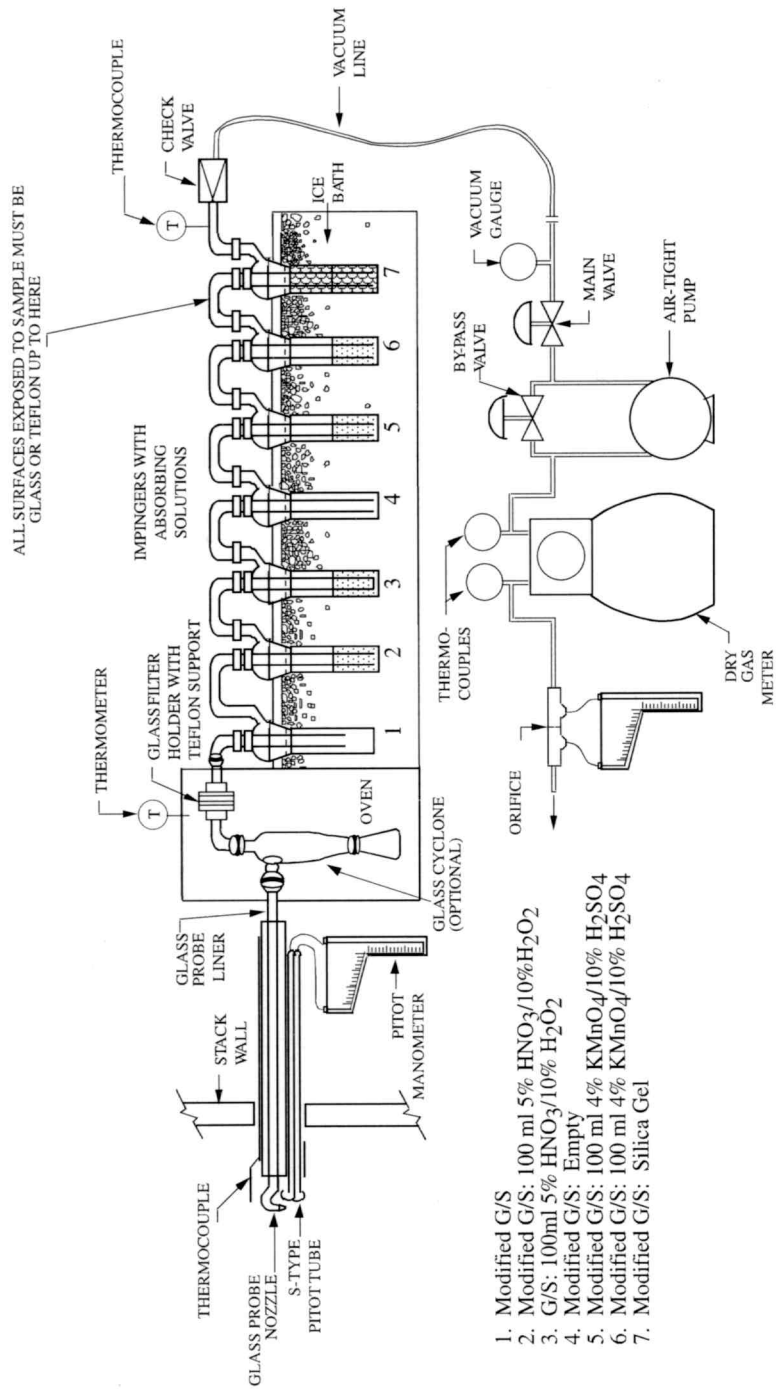


FIGURE A-22

METHOD 0060 MULTI-METALS SAMPLING TRAIN

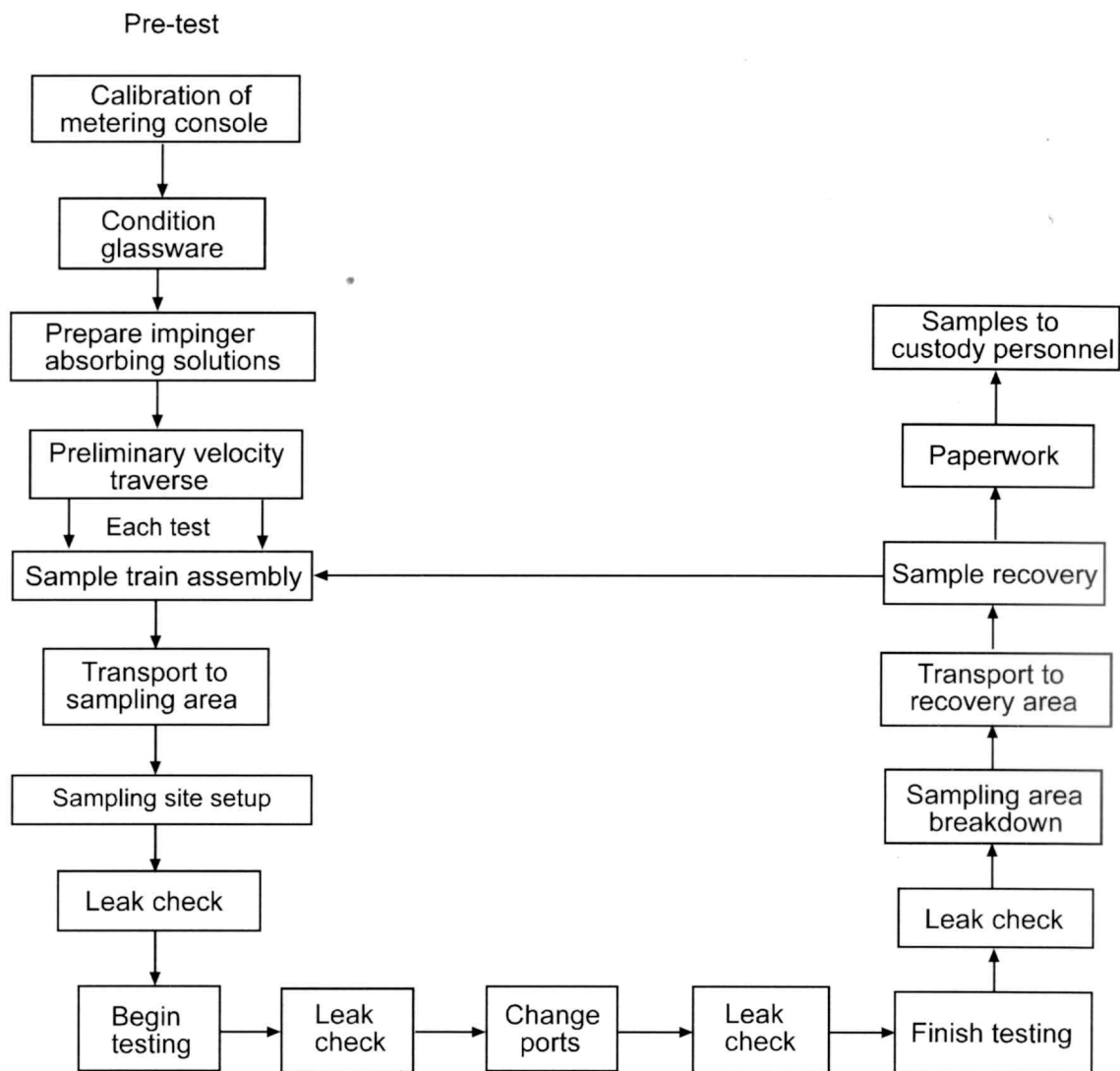


FIGURE A-23

METHOD 0060 METHODS OVERVIEW

TABLE A-7 SAMPLE TRAIN COMPONENT CHECKLIST FOR METHOD 0060

Probe Nozzle	- Quartz or Borosilicate Glass
Probe Liner	- Borosilicate glass or quartz tubing
Probe Sheath and Heater	- Stainless Steel (316) probe sheath capable of maintaining the exit or the probe at $120^{\circ}\text{C} \pm 12^{\circ}\text{C}$
Pitot tube	- Type S (as per EPA Method 2)
Differential pressure gauge	- Inclined manometer (as per EPA Method 2)
Filter holder	- Borosilicate glass with a Teflon [®] frit filter support and gasket
Filter	- Quartz fiber or glass fiber filter without organic binder, 99.95% efficient to 0.3 micron dioctyl phthalate smoke particles
Filter heating system	- Capable of maintaining the filter holder at $120^{\circ}\text{C} \pm 12^{\circ}\text{C}$
Crushed Ice	- 10-50 pounds depending on the ambient temperature
Impinger train	- Three (3) to seven (7) 500 ml impingers, connected in series with leak free ground-glass joints
Metering system	- Vacuum gauge, leak-free pump, thermometers, dry gas meter, and related equipment, as shown in Figure 4-1
Barometer	- Aneroid, or other capable of measuring to within 2.5 mm Hg
Gas density equipment	- Temperature sensor and pressure gauge, and gas analyzer
Calibration/field-prep record	- A permanently bound laboratory notebook
Teflon [®] tape	- For sealing connections and capping openings.

TABLE A-8 SAMPLE RECOVERY CHECKLIST FOR METHOD 0060

Probe liner brush	- Nylon or Teflon [®] bristle non-metallic brush
Wash bottles	- Teflon [®] (3) 500 ml
Sample storage containers	- Chemically resistant, borosilicate amber and clear glass with screw-cap liners made of Teflon [®] (1000 ml, 500 ml, 250 ml) for permanganate solution and Nalgene [®] or polyethylene for all other solutions.
Petri dishes	- Glass, sealed with Teflon [®] tape for transportation of filter samples
Graduated cylinder/balance	- To measure condensed water to nearest 0.5 ml or 0.5 g
Plastic storage containers	- Screw-cap polypropylene or polyethylene for silica gel
Teflon [®] tweezers	- For filter recovery
Funnel and rubber policeman	- To aid in the transfer of silica gel
Funnels	- Glass, to aid in sample recovery
Filters	- Quartz fiber or Glass fiber, without organic binder, 99.95% efficient
Crushed ice	- For cooling train during the run and for transport of samples
Stopcock grease	- Solvent-insoluble, heat-stable silicone grease
Silica gel	- Indicating type, 6-16 mesh
Impinger solutions	- 5% HNO ₃ /10% H ₂ O ₂ 4% KMnO ₄ /10% H ₂ SO ₄
Water	- Distilled organic-free (Type II)

the first traverse point (if traversing is to be performed) and begin sampling. Stack gas velocity is measured at The probe will be constructed of a single, heated, stainless steel sheath enclosing a borosilicate glass or quartz glass liner and a quartz or glass nozzle. The temperature of the probe will be monitored by a thermocouple placed under the probe heater in the back half of the probe. The nozzle is tapered to meet sharp-edged nozzle requirements.

The sampling probe is connected directly to the filter assembly. The filter assembly is installed in a temperature controlled oven to maintain a temperature of $248^{\circ}\text{F} \pm 25^{\circ}\text{F}$. The filter assembly is constructed of borosilicate glass. The filter support is constructed of Teflon[®]. The filters must exhibit at least 99.95% removal efficiency on $0.3\text{ }\mu\text{m}$ dioctyl phthalate smoke particles. Following the filter assembly will be a series of seven (7) impingers positioned in an ice bath. The first impinger will be empty to collect condensate from the gaseous sample. High moisture content is expected to be present during the “initial heating” and “boil-off” phases of the remediation. If an appreciable volume of condensate is expected to be collected, the first impinger may be enlarged to collect the condensate from the gas. The first impinger is modified without a stem so the sample gas does not bubble through the collected condensate. The other impingers in the sampling train are of modified Greenberg-Smith (GS) design. The second and third impingers will each be charged with 100 ml of 5% HNO_3 /10% H_2O_2 solution. The fourth impinger will be empty. The fifth and sixth impingers will each be charged with 100 ml of 4% KMnO_4 /10% H_2SO_4 solution. The seventh impinger will contain a known weight of silica gel. Impinger weights and contents will be recorded on an impinger weigh sheet, which stays with the impinger set during the test. All connections within the train will be glass or Teflon[®]. The impingers are followed by a meter box containing a pump, dry gas meter, and a calibrated orifice meter. Assembly procedures are shown in Figure A-24.

A hand calculator is used to calculate the orifice meter reading corresponding to isokinetic flow.

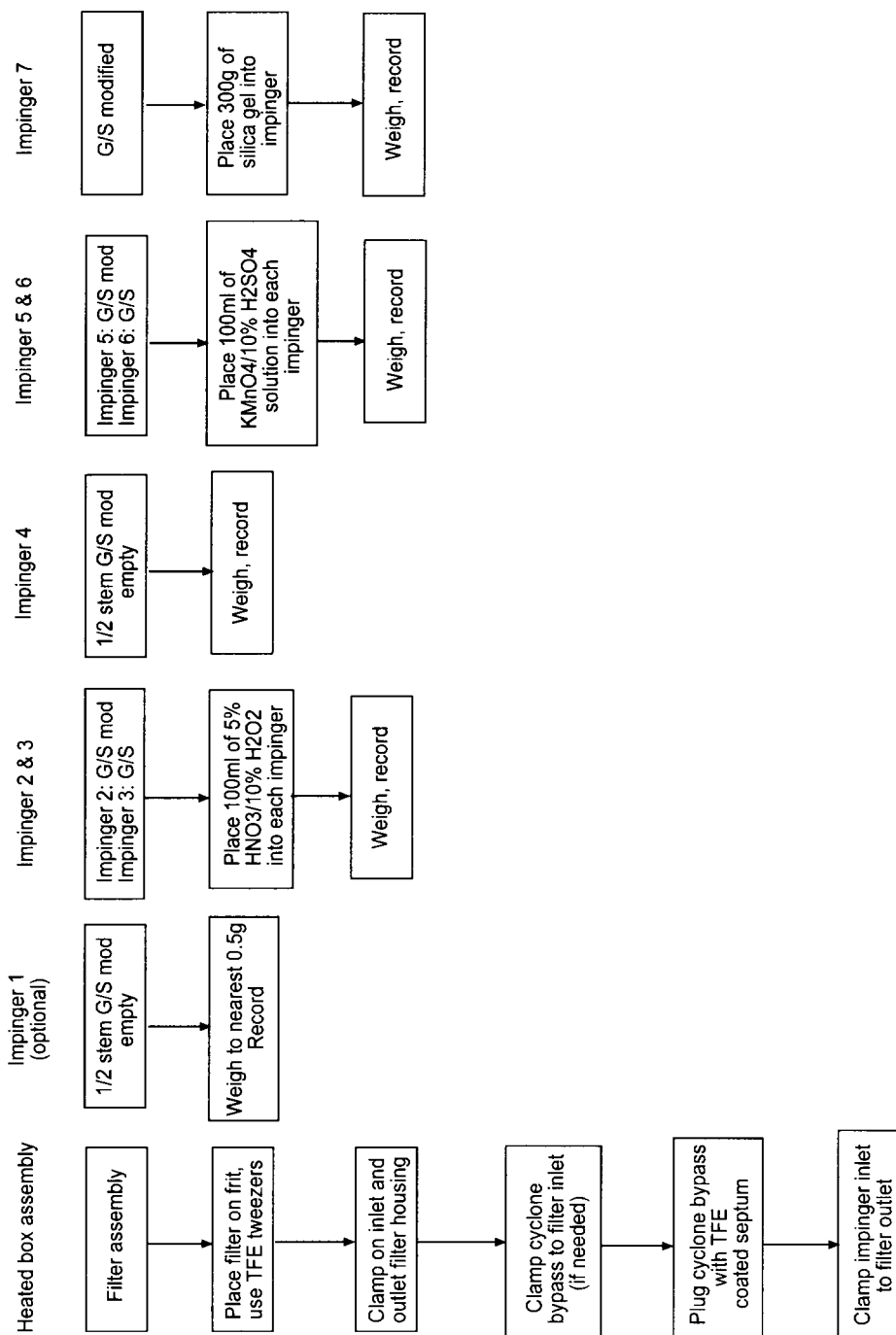


FIGURE A-24 METHOD 0060 SAMPLE TRAIN ASSEMBLY PROCEDURE

All field data will be recorded using black ballpoint pens.

The sampling train will be leak checked at the sampling site, after the train has reached sampling temperature, and prior to any sampling activities. During the run, the filter will be maintained in the temperature range of $248^{\circ}\text{F} \pm 25^{\circ}$ ($120^{\circ}\text{C} \pm 14^{\circ}$). The sampling pump will be started with the fine adjustment completely open and the coarse adjustments completely closed. The coarse adjustment will be opened slowly while the fine adjustment is being closed until a vacuum of 15 mm Hg is reached. A leak rate of no more than $0.00057 \text{ M}^3/\text{min}$ (0.02 cfm) or 4% of the sampling rate, whichever is less, is acceptable.

When sampling has been completed, as soon as the probe is withdrawn from the sampling port, the nozzle will be covered with a non-contaminating cap and the probe will be disconnected from the filter holder. The train components will be allowed to cool until they can be handled safely. All external particulate matter will then be wiped from the probe, then both ends sealed with Teflon[®] tape. Both openings of the filter holder and the impinger train will be sealed. All components of the sample train will be delivered to the recovery area. The probe is rinsed with the specified solvents into a twosome socket flask attached directly to the end of the probe or line. This reduces the possibility of sample loss during recovery. The filter is recovered first by carefully removing the filter from the housing with a pair of acid-washed polypropylene or Teflon[®] coated tweezers. Carefully transfer the filter and any particulate matter or filter fibers to a glass petri dish using an acid washed nylon or Teflon[®] bristle brush. Seal the petri dish with Teflon[®] tape. Label the petri dish containing the filter as "Container No. 1".

Taking care not to introduce dust or particulate matter on the external surfaces of the probe and nozzle into the sample, rinse the probe liner and interior surfaces on the nozzle with 100 ml of acetone. The recovery containers for the rinse liquids will be "certified-clean" 250 or 500 ml glass bottle with a

Teflon[®]-lined cap. Using a non-metallic brush, brush the interior surfaces of the nozzle and probe liner while rinsing with acetone until no visible particles are present in the final rinse. Rinse the front half of the filter housing in the same manner as the probe liner and nozzle. Label the bottle as "Container No. 2".

Rinse the probe liner, nozzle, and front half of the filter housing with 0.1N HNO₃ using the same procedure as was used for the acetone rinse. Collect the rinseate in a "certified-clean" 250 or 500 ml glass bottle with a Teflon[®]-lined cap. Do not use more than 100 ml of solution to perform the rinse. Label the bottle as "Container No. 3".

"Container No. 4" will contain the contents of impingers 1 through 3, plus the rinseates of these impingers. Prior to recovery, each impinger is weighed to determine the moisture gain to each impinger. The weight of each impinger, to 0.5 g, is entered onto the weigh sheet, which will accompany the train from assembly through recovery. If a large quantity of condensate has been collected, the contents of the impingers and the rinseates may be recovered into more than one container. Transfer the contents from each of the three impingers into a 500 ml or 1000 ml Nalgene[®] or polyethylene bottle. Then clean each of the first three impingers, the back half of the filter housing, the filter support, and all connecting glassware by thoroughly rinsing with a total of 100 ml of 0.1 N HNO₃. Add the rinseate to the contents of the impingers. Label the container(s).

Weigh Impinger 4 to 0.5 g and record the weight on the weigh sheet. The weight will be used to calculate the moisture content in the gas stream. Transfer the contents of the impinger into a 250 ml or 500 ml Nalgene[®] or polyethylene bottle. Rinse the impinger with 100 ml of 0.1 N HNO₃. Combine the rinseate in the bottle with the impinger contents. Label the sample container as "Container No. 5A".

Weigh Impingers 5 and 6 and record the weight. This information will also be used to calculate the

moisture content in the sample gas. Transfer the contents from these impingers into a 500 ml or 1000 ml amber glass bottle with a Teflon[®]-lined cap. Label the bottle as "Container No. 5B". Use a total of 100 ml of fresh acidified permanganate solution to rinse impingers 5 and 6 a minimum of three (3) times. Add the rinses to Container No. 5B. Use a total of 100 ml of water to rinse impingers 5 and 6 and the connecting glass pieces a minimum of three (3) times. Add the rinses to Container No. 5B. Make sure that any precipitated material is transferred to the container. If any residue remains in the impingers, in a hood or well ventilated area, rinse the impinger surfaces and the stem with 25 ml of 8M HCl. Transfer the rinse to a 500 ml Nalgene[®] or polyethylene bottle which contains 200 ml of water. Label the bottle as "Container 5C".

Weigh Impinger 7 and record the weight. This information will also be used to calculate the moisture content in the sample gas. Transfer the silica gel from the impinger into a 500 ml Nalgene[®] or polyethylene bottle. A rubber policeman and a funnel may be used to remove the silica gel. Note the color of the silica gel on the weigh sheet to determine if it was completely spent.

Figure A-25 shows the recovery scheme to be used for this test program. Once recovered, all sample fractions will be stored at ambient temperature until transported to the laboratory. The filters will be stored separately at ambient temperature until transported to the laboratory. All QA/QC samples as defined for the program will also be collected. These samples include all applicable reagent blanks, field blanks, filter blanks, and water blanks.

A.7 EPA Method 0050 - HCl and Cl₂ and Particulate Matter

Hydrogen chloride, Chlorine, and particulate matter in the gaseous emissions will be collected

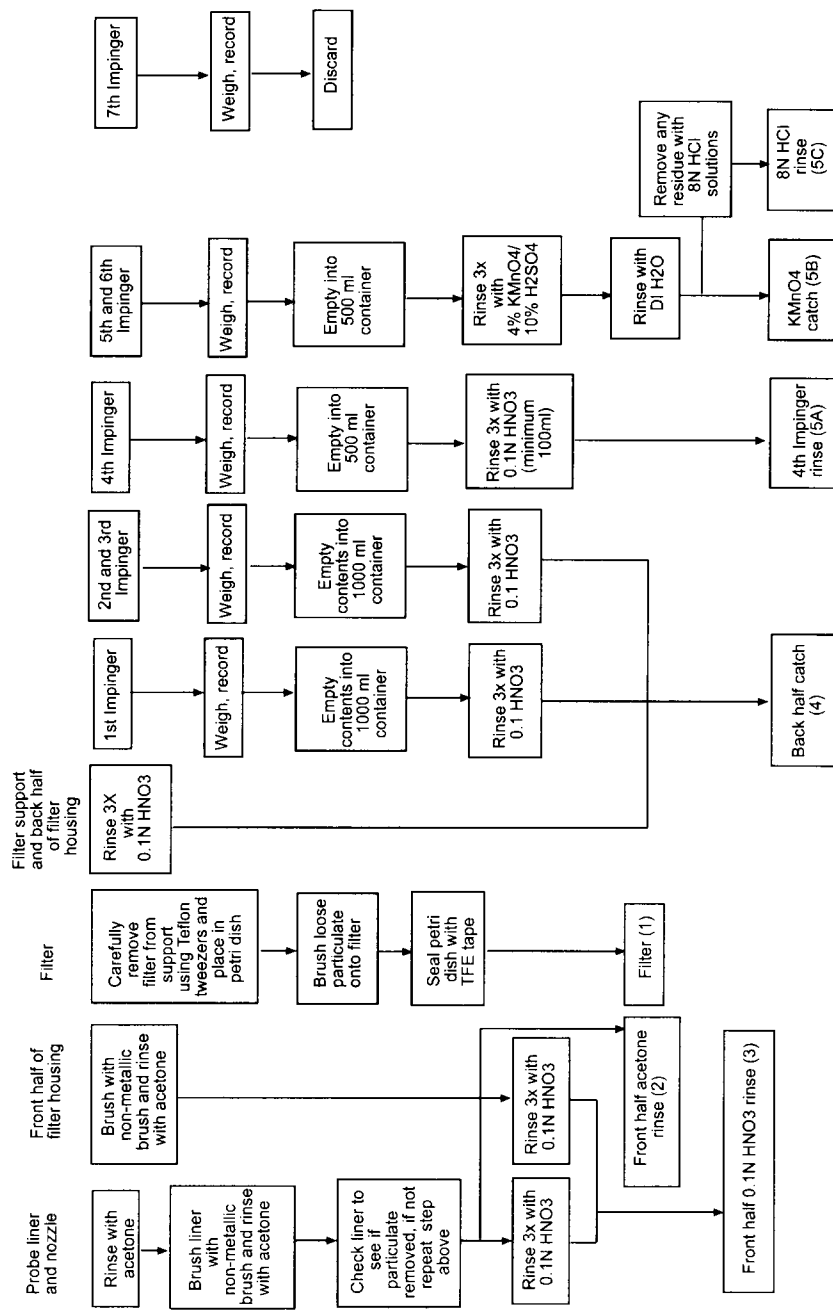


FIGURE A-25 METHOD 0060 SAMPLE RECOVERY PROCEDURE

from the H and I locations of the melter system using EPA Method 0050. This sampling method is based on EPA Method 26. Particulate matter is not expected to be present in the gas stream. Therefore, a particulate determination may not be calculated from this sampling train. If during collection of gas samples using the other methodologies particulate matter is present, then a particulate determination will be performed. The collected samples will be transported to the analytical laboratory for preparation and analysis. Gaseous and particulate pollutants will be withdrawn from the emission source at an isokinetic rate and collected in the sampling train illustrated in Figure A-26. If the minimum stack dimension requirements are not met, the stack will not be traversed, but the probe nozzle will be positioned in the centroid of the duct for sampling. Particulate emissions are collected in the probe and on the heated filter. Gaseous emissions are collected in a series of chilled impingers. An overview of the sampling method is provided in Figure A-27.

Assembly of the sampling train begins in the recovery area. Tables A-9 and A-10 list the equipment needed to assemble and recovery the sampling train. During the assembly of the train, a visual inspection of each of the sampling train components and equipment is performed. Glassware will be checked for cracks and chips, all connections will be cleaned to ensure good seals. Fittings and ferrules will be inspected for wear. Tubing are inspected for wear and flexibility. All defective equipment will be replaced. All components of the train will be covered with aluminum foil during the assembly process. No silicon grease will be used on any ground glass joints.

All glassware will be thoroughly cleaned prior to use. All glassware will be first washed in hot water with a non-phosphorus laboratory detergent. The glassware is then rinsed with tap water and rinsed three times with deionized water. All glassware is then placed on a Teflon[®] or polypropylene rack and allowed to air dry in an organic, solvent-free area. After drying, all

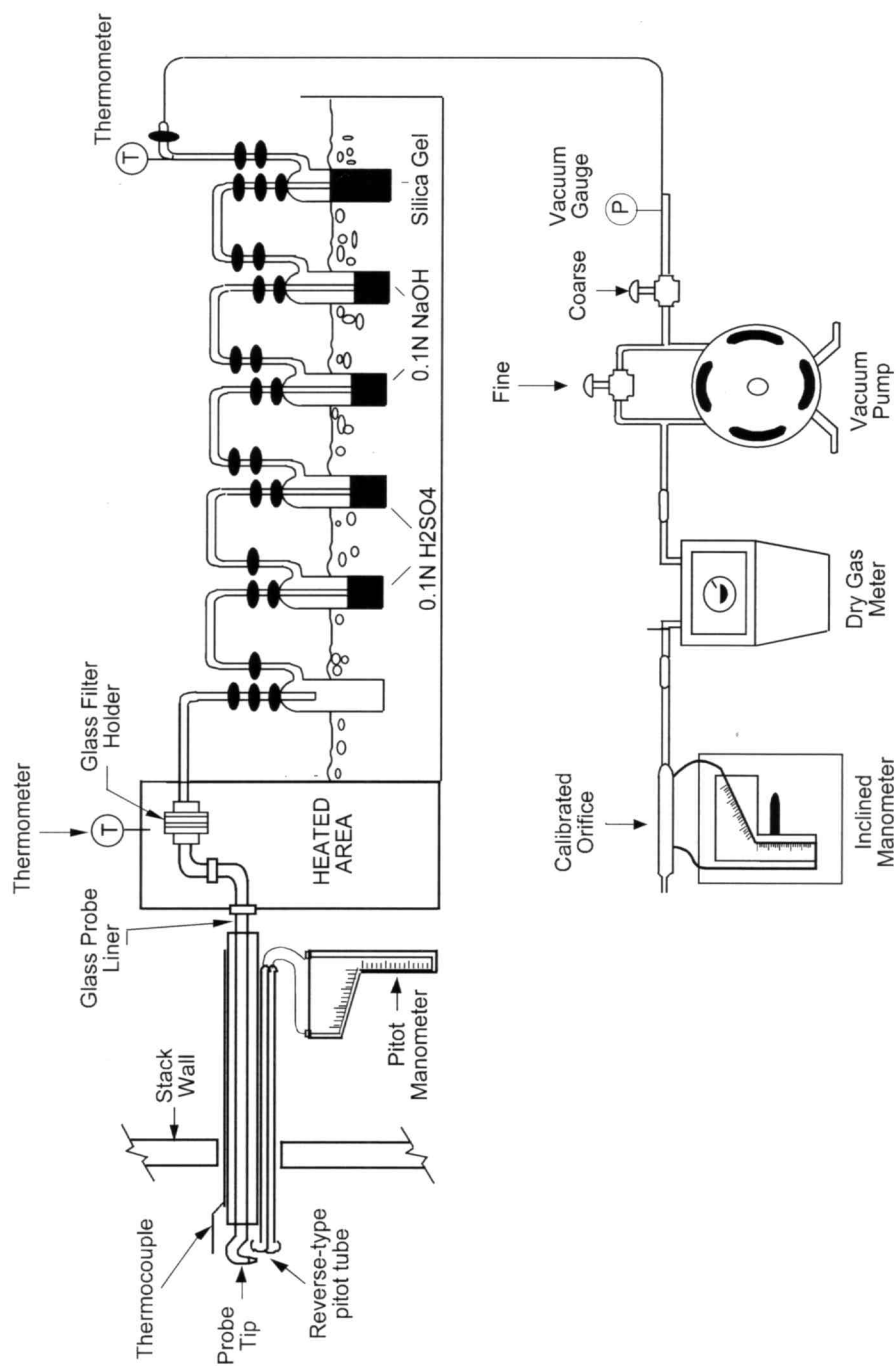


FIGURE A-26 METHOD 0050 HCl and Cl₂ SAMPLE TRAIN SCHEMATIC

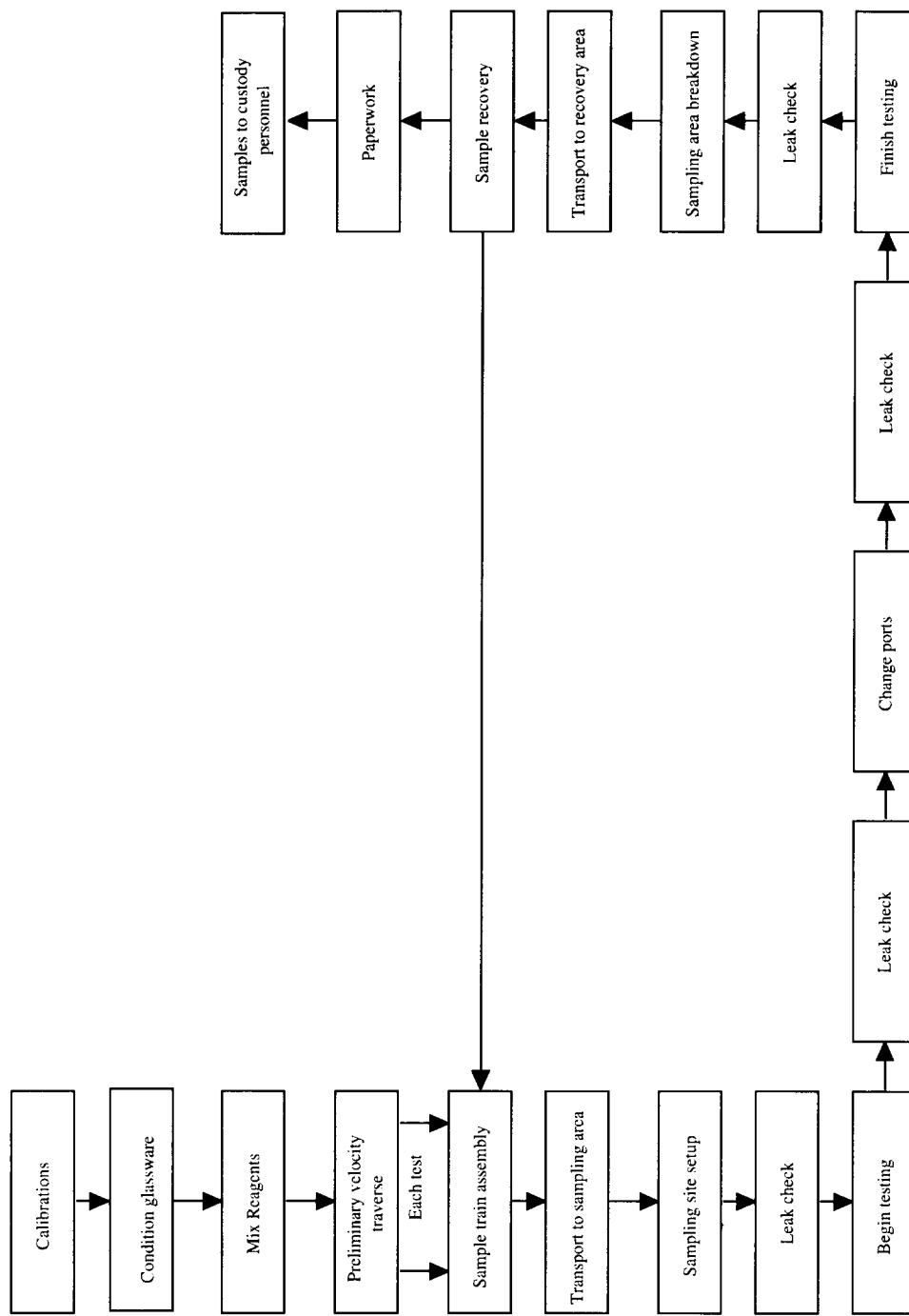


FIGURE A-27

METHOD 0050 METHOD OVERVIEW

TABLE A-9 SAMPLE TRAIN COMPONENT CHECKLIST FOR METHOD 0050

Probe Nozzle	- Quartz or Borosilicate Glass
Probe Liner	- Borosilicate glass or quartz tubing
Probe Sheath and Heater	- Stainless Steel (316) probe sheath capable of maintaining the exit or the probe at $120^{\circ}\text{C} \pm 12^{\circ}\text{C}$
Pitot tube	- Type S (as per EPA Method 2)
Differential pressure gauge	- Inclined manometer (as per EPA Method 2)
Glass Cyclone	- Borosilicate glass, used for high particulate sample stream (optional)
Filter holder	- Borosilicate glass with a Teflon [®] frit filter support and gasket
Filter	- Quartz fiber or Teflon [®] mat
Filter heating system	- Capable of maintaining the filter holder at $120^{\circ}\text{C} \pm 12^{\circ}\text{C}$
Crushed Ice	- 10-50 pounds depending on the ambient temperature
Impinger train	- Five or six (5 or 6) 500 ml impingers, connected in series with leak free ground-glass joints
Metering system	- Vacuum gauge, leak-free pump, thermometers, dry gas meter, and related equipment, as shown in Figure 4-1
Barometer	- Aneroid, or other capable of measuring to within 2.5 mm Hg
Gas density equipment	- Temperature sensor and pressure gauge, and gas analyzer
Calibration/field-prep record	- A permanently bound laboratory notebook
Ascarite tube	- Used to condition ambient air

TABLE A-10 SAMPLE RECOVERY CHECKLIST FOR METHOD 0050

Probe liner brush	- Nylon or Teflon® bristle brush with stainless steel wire or Teflon® handle, Teflon® extensions at least as long as the probe may be used
Nozzle brush	- Nylon or Teflon® bristle brush with stainless steel wire or Teflon® handle
Wash bottles	- Teflon® (3) 500 ml
Sample storage containers	- Chemically resistant, Nalgene® or polyethylene bottles; 1000 ml, 500 ml, 250 ml Wide mouth “Certified” glass bottles with Teflon®-lined caps; 500 ml, 250 ml
Petri dishes	- Glass, sealed with Teflon® tape for transportation of filter samples
Graduated cylinder/balance	- To measure condensed water to nearest 0.5 ml or 0.5 g
Plastic storage containers	- Screw-cap polypropylene or polyethylene for silica gel
Funnel and rubber policeman	- To aid in the transfer of silica gel
Funnels	- Glass, to aid in sample recovery
Filters	- Pre-weighed Quartz fiber or Teflon® mat, desiccated to a constant weight
Teflon® tweezers	- For filter recovery
Crushed ice	- For cooling train during the run and for transport of samples
Silica gel	- Indicating type, 6-16 mesh
Impinger solutions	- Sulfuric Acid (0.1 N), H ₂ SO ₄ , used as HCl absorbing solution Sodium Hydroxide (0.1N) NaOH, used as Cl ₂ absorbing solution
Reagent Water	- Distilled organic-free (Type II)
Acetone	- Residue grade for particulate determination
Sodium Thiosulfate	- Crystals for sample preservation

components will be sealed with glass caps and Teflon[®] tape. All sample recovery containers will be purchased as “Certified”, indicating the containers have been cleaned to U.S. EPA Contract Laboratory Program analyte specifications. Sampling filters and impinger solutions will be prepared as specified in the methodology.

The probe will be constructed of a single, heated, stainless steel sheath enclosing a borosilicate glass or quartz glass liner and a quartz or glass nozzle. The temperature of the probe will be monitored by a thermocouple placed under the probe heater in the back half of the probe. The nozzle is tapered to meet sharp-edged nozzle requirements.

The sampling probe is connected directly to the filter assembly. The filter assembly is installed in a temperature controlled oven to maintain a temperature of $248^{\circ}\text{F} \pm 25^{\circ}\text{F}$. The filter assembly is constructed of borosilicate glass. The filter support is constructed of Teflon[®]. The quartz glass or Teflon[®] mat filters must be labeled and pre-weighed. Following the filter assembly will be a series of six (6) impingers positioned in an ice bath. The first impinger is optional and will be used during the “initial heating” and “boil-off” phases, since high moisture concentrations will be present in the gas stream. The first impinger it will contain 50 ml of 0.1N H_2SO_4 to collect condensate from the gaseous sample. The impinger is modified with a short stem so the sample gas does not bubble through the collected condensate. The other impingers in the sampling train are of modified Greenberg-Smith (GS) design. The second and third impingers will each be charged with 100 ml of 0.1N H_2SO_4 solution. The fourth impinger and fifth impinger will each contain 100 ml of 0.1N NaOH solution. The sixth impinger will contain a known weight of silica gel. Impinger weights and contents will be recorded on an impinger weigh sheet, which stays with the impinger set during the test. All connections within the train will be glass or Teflon[®]. The impingers are followed by a meter box containing a pump, dry gas meter, and a calibrated orifice meter. Assembly procedures are shown in Figure A-28.

Method 26 Train Assembly

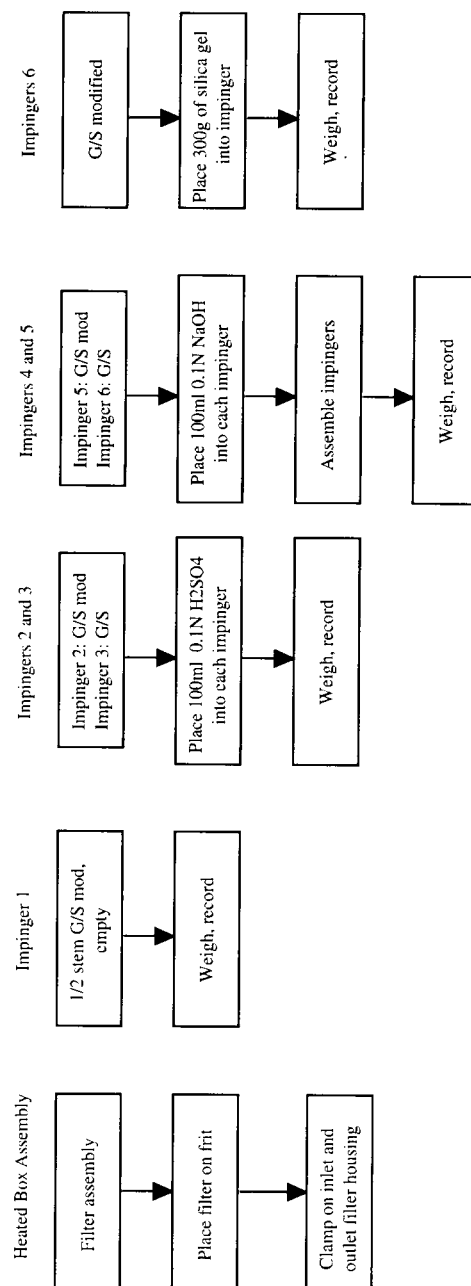


FIGURE A-28 METHOD 0050 SAMPLE TRAIN ASSEMBLY PROCEDURE

Following assembly of the HCl/Cl₂ train, the train is transported to the sampling location. The sampling port is cleaned to prevent contamination from particulate matter present in the port cover. The operator then informs the crew chief and awaits instructions to position the probe at the first traverse point (if traversing is to be performed) and begin sampling. Stack gas velocity is measured at each traverse point with an S-type pitot tube. A hand calculator is used to calculate the orifice meter reading corresponding to isokinetic flow. Since the duct work at each sampling location is less than 12 inches in diameter, Method 1A will apply. Traversing the duct during sampling will not be performed. All field data will be recorded using black ballpoint pens.

The sampling train will be leak checked at the sampling site, after the train has reached sampling temperature, and prior to any sampling activities. During the run, the filter and optional cyclone will be maintained at a minimum of 248°F ± 25° (120°C ± 14°). The sampling pump will be started with the fine adjustment completely open and the coarse adjustment completely closed. The coarse adjustment will be opened slowly while the fine adjustment is being closed until a vacuum of 15 mm Hg is reached. A leak rate of no more than 0.00057 M³/min (0.02 cfm) or 4% of the sampling rate, whichever is less, is acceptable.

When sampling has been completed, as soon as the probe is withdrawn from the sampling port, the nozzle will be covered with aluminum foil and the train components will be allowed to cool until they can be handled safely. All external particulate matter will then be wiped from the probe, then remove the probe and seal both ends sealed with aluminum foil. Both openings of the filter holder and the impinger train will be sealed. All components of the sample train will be delivered to the recovery area. The filter is recovered first by carefully removing the filter from the housing with a pair of Teflon[®] coated tweezers. Carefully transfer the filter and any particulate matter or filter fibers to a glass petri dish using a nylon or Teflon[®] bristle brush. Seal the petri dish with Teflon[®] tape. Label the filter as "Container No. 1".

Taking care not to introduce dust or particulate matter on the external surfaces of the probe and nozzle into the sample, rinse the probe liner, the front half of the filter housing, the cyclone (if used), and the interior surfaces on the nozzle with 100 ml of acetone. The recovery containers for the rinse liquids will be "certified-clean" 250 or 500 ml glass bottles with a Teflon®-lined cap. Using a non-metallic brush, brush the interior surfaces of the nozzle and probe liner while rinsing with acetone until no visible particles are present in the final rinse. Rinse the front half of the filter housing in the same manner as the probe liner and nozzle. Label the bottle as "Container No. 2".

Weigh each of the impingers and record their weight on the weigh sheet which accompanied the sample train from assembly until returned for recovery. This information is required to calculate the moisture concentration in the gas sampled from the source.

Transfer the contents of Impingers 1 through 3 to a 1000 ml Nalgene® or polyethylene bottle. Rinse these impingers and the connecting glassware with deionized water. Add the rinse to the contents in the sample bottle. Rinse the filter support and the back half of the filter housing with deionized water. Collect the rinse and transfer to the contents of the sample bottle. Label the bottle as "Container No. 3". This sample fraction will be used to determine HCl concentration.

Transfer the contents of Impingers 4 and 5 to a 1000 ml Nalgene® or polyethylene bottle. Rinse these impingers and the connecting glassware with deionized water. Add the rinse to the contents in the sample bottle. Add 100 mg of Sodium thiosulfate crystals to the sampling container to scavenge the OCl⁻ ions in solution. Label the sample bottle as "Container No. 4". This sample fraction will be used to determine Cl₂ concentration.

Mark the fluid level on the outside of each sample bottle using a permanent marker. This mark will be

used to check for leakage during shipment. Seal the cap with Teflon[®] tape.

Weigh Impinger 6 and record the weight. This information will also be used to calculate the moisture content in the sample gas. Transfer the silica gel from the impinger into a 500 ml Nalgene[®] or polyethylene bottle. A rubber policeman and a funnel may be used to remove the silica gel. Note the color of the silica gel on the weigh sheet to determine if it was completely spent.

Figure A-29 shows the recovery scheme to be used for this test program. Once recovered, all sample fractions will be stored at ambient temperature until transported to the laboratory. The filters will be stored separately at ambient temperature until transported to the laboratory.

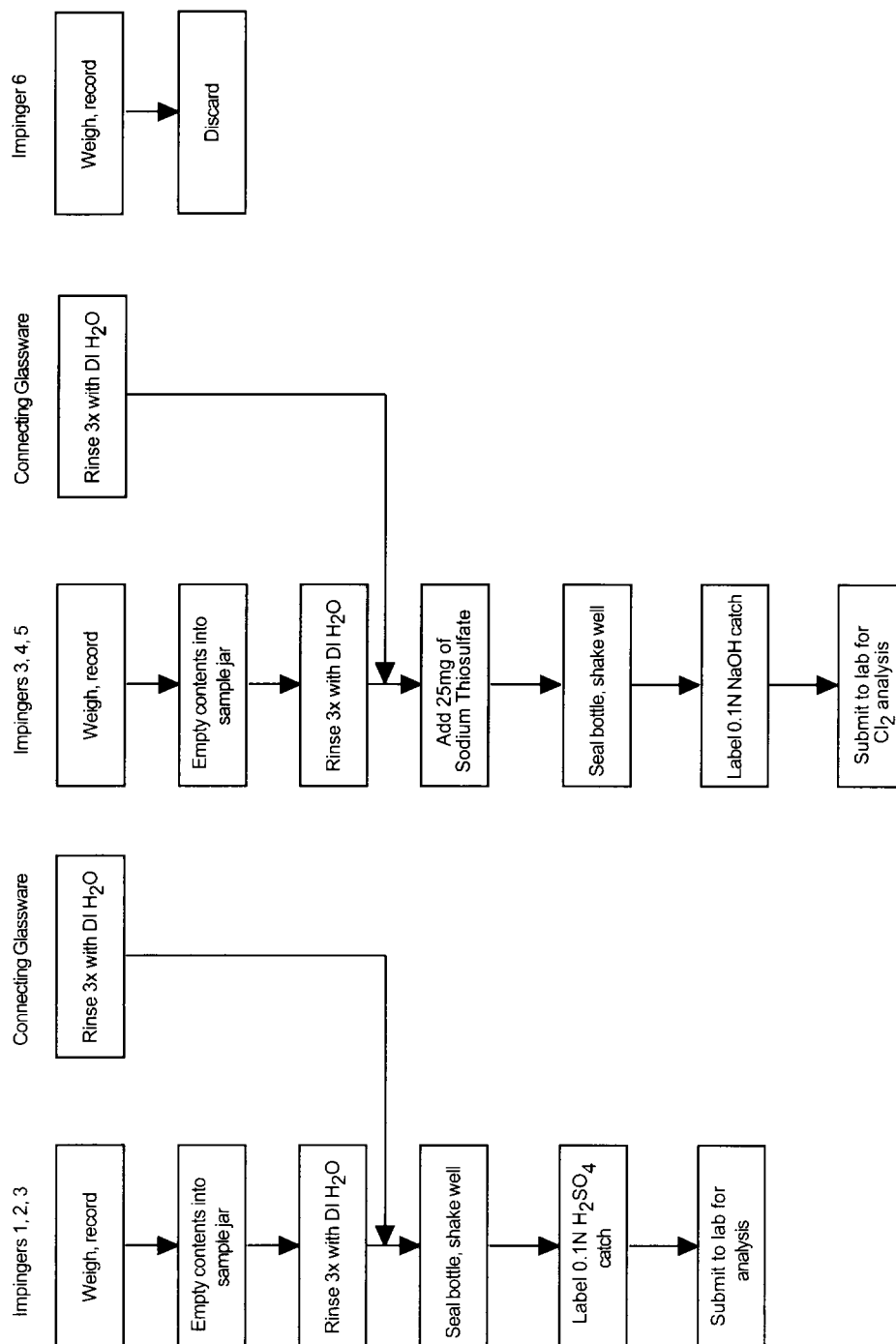


FIGURE A-29METHOD 0050 SAMPLE RECOVERY PROCEDURE

APPENDIX B

ANALYTICAL METHODS

Appendix B

Analytical Methods

The analytical procedures used by the laboratory will follow the referenced EPA methodologies with the following noted exceptions and deviations.

B.1 Volatile Organic Compounds

Samples for VOC analysis will only be collected during the vitrification phase of the project. Dried sediment will be sampled for VOCs (Method 5035/8260B) by compositing several samples collected from the batches of sediment and flux that are mixed for processing in the melter. The purpose of analyzing the dried sediment samples for VOCs is to demonstrate the absence VOCs in the sediment. Each of the flue gas samples collected for VOC analysis at sampling location H or I (Figure 4-2) consists of two unique fractions. Each fraction must be analyzed by a specific method. The gaseous fraction of the sample, collected on the VOST cartridges, will be analyzed according to Method 5041A/8260B. The aqueous sample (consisting of condensate collected in a trap between the second and third VOST cartridges) will be analyzed by Method 5030B/8260B.

B.1.1 Method 5041A/8260B

The collection of the VOST cartridges for VOCs is a secondary concern to the vitrification phase of the test program. Halogenated species are not expected to be generated during the thermal decomposition of PCBs during the vitrification process. Samples will be analyzed for VOCs to validate this hypothesis. If VOCs are detected in the VOST cartridges, samples of dried sediment should show that VOCs are not present in the sediment.

B.1.2 Method 5030B/8260B

The condensate fraction collected from the Method 0031 sampling trains will be analyzed according to Method 5030B/8260B. Method 5030B/8260B utilizes purge-and-trap extraction of the volatile components from the sample, then gas chromatography/mass spectrometry (GC/MS) for separation and quantitation. Dried sediment will also be sampled for VOCs by compositing several samples collected from the batches of sediment and flux that are mixed for processing in the melter. The purpose of analyzing the dried sediment samples for VOCs is to demonstrate the absence VOCs in the sediment.

B.2 Semivolatile Organic Compounds

SVOCs, particularly the fate of PCBs during both the drying process and the vitrification process, are the critical parameters of this program. Samples will be collected for PCB congeners using EPA Method 23. The Method 23 samples collected during the second phase of the project will be analyzed for PCDDs and PCDFs (in addition to PCBs) to determine if these compounds are formed in the flue gas from the vitrification process. SVOCs will also be collected using a Method 0010 train, which will be analyzed for semivolatile principal organic hazardous compounds (POHC).

B.2.1 Polychlorinated Biphenyls

Samples will be analyzed for PCBs using Method 680. In addition to the coplanar PCB congeners specified in Method 680, several additional congeners will also be target analytes to determine the TE of the glass melter technology.

The collection of the Method 23 sample train for PCBs is critical to the test program. The main concern of this testing program is to verify that PCBs are not liberated during either the drying process or the vitrification process. PCBs are technical mixtures historically produced for industrial purposes, and as such, they consist of many congeners and chlorinated precursors. Method 680 will be used to quantify

the respective congeners as opposed to quantifying the specific Aroclors present in the sediments. The respective congeners may be thermally decomposed at different rates. Therefore, the fate of several prominent congeners, in addition to the coplanar congeners, will be determined.

B.2.2 Polychlorinated Dibenzo-p-Dioxins and Polychlorinated Dibenzofurans

Samples will be analyzed for PCDDs and PCDFs according to Method 23/Method 8290. The method utilizes HRGC/HRMS. Sampling will be performed to assure that dioxins and furans are not formed in the emissions from the melter or the dryer.

B.2.3 Principle Organic Hazardous Compounds

During the vitrification phase of the project, samples collected for SVOCs will be analyzed according to Method 8270C. The analytical method utilizes high resolution gas chromatography/low resolution mass spectrometry (HRGC/LRMS). SVOCs will be analyzed to determine which, if any, are liberated from the melting process or are formed from the thermal decomposition of the PCBs.

B.3 Metals

Air, water and soil samples submitted to the laboratory for analysis of metals and mercury will be analyzed according to Method 6010B and Method 7470A/7471A. Method 6010B uses an inductively coupled plasma-atomic emission spectrometer to quantify metals in the collected sediment, condensate, and flue gas samples. Method 7470A is used to quantify mercury in aqueous samples, and Method 7471B is used to quantify mercury in solid samples. Method 7470A/7471B uses a cold-vapor atomic absorption technique to quantify mercury. Samples for metals will be collected during both phases of the project to determine during which phase of the process metal species may be volatilized and to determine the extent of volatilization.

Sediment samples, aggregate samples, and water samples collected during the various stages of the process will be prepared according to the matrix-applicable methodologies referenced in SW-846. Sediments samples and glass aggregate samples will be digested according to Method 3050B. Glass aggregate samples to be tested for leachable metals will be prepared using the designated method (ASTM or SPLP), followed by digestion of the resulting leachate using SW-846 Method 3010A. All aqueous samples will be digested using Method 3010A. The resulting digestates from all matrices will be analyzed using Method 6010B, Inductively Coupled Plasma-Atomic Emission Spectrometry (ICP) for all metals except mercury.

Interference check samples will be analyzed on the ICP as noted in Method 6010B. A Method Blank, a Laboratory Control Sample, and a Laboratory Matrix Spike sample will be prepared for each digestion batch as noted in the methodology. If interferences are noted in the analyzed digestates, which cannot be addressed using interelement corrections, then the digestates will be diluted and reanalyzed. If the interferences are still present, then one sample per matrix will be analyzed to determine the quantification levels applicable to the respective matrix.

Aqueous samples (including the leachates) for mercury will be prepared and analyzed using Method 7470A. All sediment and aggregate samples for mercury will be prepared and analyzed using Method 7471A. A Method Blank, a Laboratory Control Sample, and a Laboratory Matrix Spike sample will be prepared for each digestion batch as noted in the methodology.

B.4 Hydrogen Chloride and Chlorine

Samples for hydrogen chloride and chlorine will be collected only during the second phase of the project. Hydrogen chloride and chlorine samples submitted to the laboratory will be analyzed according to EPA Method 26 (Method 9057). The analytical method utilizes an ion chromatograph

Section: B.0
Revision: 2
Date: 7/24/01
Page: B-5

with an electrolytic conductivity detector. Each impinger fraction will be analyzed separately in duplicate, as required by the method. The chloride ion concentration quantified in the front half of the sample train will be calculated as hydrogen chloride. The chloride ion concentration quantified in the back half of the sample train will be calculated as chlorine.

APPENDIX C

CALIBRATION PROCEDURES

Appendix C

Calibration Procedures

Prior to analysis of any sample, each of the analytical instruments must be calibrated. Calibration procedures, the frequency of continuing calibration verification, and the criteria for evaluating the calibration data are described in the EPA analytical methods. Calibration data will be recorded in the instrument logbook and referenced to the standards preparation log to identify the source and method of preparation of the standard solutions used.

C.1 Volatile Organic Compounds

After collection of samples for determination of VOCs by Method 0031, analysis is performed using an integrated two-step process. The first step, sample introduction, is used to (1) revolatilize the organic compounds from the adsorbent cartridges or the condensate samples collected in the field, (2) concentrate the volatile compounds, and (3) inject them into the GC/MS system. The second step, chromatographic and mass spectral analysis, separates, identifies, and quantifies the VOCs.

Two different sample introduction methods are required to analyze the components of the Method 0031 sampling train. Method 5041A is used to thermally desorb the “trapped” VOCs from the sorbent cartridges through a condensate trap, and then collect them on another adsorbent trap, prior to injection into the gas chromatography system. The condensate trap removes excess water from the collected sample. Method 5030B is used to dynamically strip the VOCs from the aqueous matrix and collect them on an adsorbent cartridge for injection into the GC/MS system.

This section summarizes the two sample introduction procedures and the calibration procedure for Method 8260B.

C.1.1 Method 5041A

Method 5041A is the sample introduction procedure used to transfer the VOCs collected on the Method 0031 sorbent cartridges into the gas chromatographic system. The sorbent cartridges are placed in an oven, where they are thermally desorbed by heating and purging with helium. The gaseous effluent from the cartridges is bubbled through volatile-free water to condense water trapped on the cartridges. The volatiles in the effluent are then collected on an analytical sorbent trap. After the desorption cycle is complete, the analytical sorbent trap is backflushed and thermally desorbed to transfer the VOCs into the gas chromatographic system.

Once the temperatures and flow are set for the desorption unit, no calibration of the unit is performed, per se. The desorption unit is used to introduce the appropriate calibration standards to the GC/MS system. Any problems with the operation of the system would be detected during the calibration procedure or noticed in the recovery of the surrogate and internal reference standards, which are added to every sample, blank, and reference standard prior to analysis.

C.1.2 Method 5030B

Method 5030B is the sample introduction procedure used to dynamically strip the VOCs from the condensate of the Method 0031 sampling train into the gas chromatographic system. A 5-mL (or 25-mL) aliquot of sample is transferred to a fritted chamber, where the VOCs in the water are dynamically stripped by purging the water with helium. VOCs stripped from the matrix are then collected on an analytical sorbent trap. After the purging cycle of the water is complete, the analytical sorbent trap is backflushed and thermally desorbed to transfer the VOCs into the gas chromatographic system.

Once the temperatures and gas flow are set for the purging unit, no calibration of the unit is performed. The unit is an integral part of the GC/MS system and used to introduce the appropriate calibration standards.

C.1.3 Method 8260B

Method 8260B utilizes a GC separation of the VOCs with detection using a MS. The MS ionizes the compounds as they enter the detector in unique fractionation patterns. The resulting spectra is then compared against calibration or library reference spectra. This method is discussed in further detail below.

Preparation and Supply of Sampling Media

Kemron will supply two thermally conditioned Tenax[®]-GC and one thermally conditioned Anasorb[®]-747 sorbent cartridges, as well as a 40-mL VOC vial for each sampling train. Three 40-minute sampling trains yield one sampling run. The laboratory will also supply trip blanks for each shipment and a temperature blank for each cooler. At a minimum, two 1-L bottles of volatile-free water will be supplied by the laboratory for use a field blanks and other field quality control.

Samples to be Collected

Samples for VOCs will be collected only during the second phase of this program. For each sample train, three adsorbent cartridges and one condensate fraction will be collected. Nine samples, each consisting of three sample trains, will be collected from the flue during the vitrification phase of the project. Kemron will provide the following sampling items for each train:

<u>Item</u>	<u>Number per train</u>
Tenax [®] -GC Cartridge	two per sample train
Anasorb [®] -747 Cartridge	one per sample train
40-mL VOC vial	one per sample train (for the condensate)

Sample Preparation of the Adsorbent Cartridges

The cartridge samples will be prepared for analysis as specified in EPA Method 5041A/8260B. The tubes may either be analyzed separately or combined. The cartridge to be analyzed will be placed in the thermal desorption module and spiked with a surrogate standard spiking solution. In the condensate removal vessel, an aliquot of volatile-free water will be spiked with internal standard spiking solution immediately prior to analysis.

Sample Preparation of the Condensates

The aqueous condensate samples will be prepared for analysis as specified in EPA Method 5030B/8260B. An aliquot of sample will be spiked with a surrogate standard spiking solution and internal standard spiking solution immediately prior to analysis. Table C-1 lists the components of the surrogate standard spiking solution and the internal standard spiking solution.

The internal standards and surrogate standards are selected for two reasons. First, the compounds are either deuterated (contain a deuterium atom) and/or fluorinated (contain a fluorine atom), which make them highly unlikely to be found at a sufficient concentration in the environmental system under study.

Second, the isotopic analogs (deuterated compounds) of the analytes of interest are expected to have similar behavior to the non-labeled compounds, thereby allowing correlation between the species.

TABLE C-1
STANDARDS FOR METHOD 8260B

Surrogate Standards
Toluene-d ₈
4-Bromofluorobenzene
1,2-Dichloroethane-d ₄
Internal Standards
1,4-Dichlorobenzene-d ₄
Chlorobenzene-d ₅
1,4-Difluorobenzene

Gas Chromatograph/Mass Spectrometer Calibration

At the beginning of each 12-hour analytical sequence, before any calibration standards or samples are analyzed, 50 nanograms (ng) of 4-bromofluorobenzene (BFB) will be injected into the GC/MS. Mass spectral intensities for BFB will be generated and these intensities will be used to verify that the mass assignments and the relative ion intensities meet the criteria of the methodology. If the criteria are met, then calibration standards may be injected (whether an initial five-level calibration curve or a single continuing calibration standard).

Calibration standards for each target compound will be generated from certified reference standards.

A minimum of five calibration levels for each target compound will be generated prior to the analysis of any of the samples associated with this project are analyzed.

Once the instrument has met the calibration criteria of the methodology, a method blank is analyzed prior to any samples. The method blank verifies that the analytical system is free of interferences and contamination prior to the analysis of any samples.

Sample Analysis

Once the analytical system is calibrated and the method blank criteria have been met, samples may be analyzed. The VOST tubes may be analyzed either individually or in pairs. Surrogate spiking standards are added to the tubes, and internal standards are added to the water module. The tubes are thermally desorbed into the GC/MS system for analysis. The samples may be analyzed until the 12-hour calibration window has expired. Then the calibration of the system is verified and another 12-hour calibration window begins. If the concentration of an analyte in a sample exceeds the calibration range of the instrument, a splitter may be added to the thermal desorption module to allow a smaller mass of trapped organics to be transferred to the analytical system.

Once the analytical system for the condensate samples is calibrated and the method blank criteria has been met, the condensate samples may be analyzed. A 5-mL (or 25-mL) aliquot of the condensate water samples should be spiked with the surrogate standard solution and the internal standard solution, then loaded into the purge-and-trap autosampler. The samples will then be analyzed sequentially during a 12-hour calibration window. If the concentration of an analyte in a sample analysis exceeds the calibration range of the instrument, the condensate sample is diluted with volatile-free water and a 25-mL aliquot of the diluted sample will be spiked with the surrogate standard solution and internal standard solution before being analyzed.

Once the major components in each of the sample fractions have been identified, quantitative analyses of those compounds will be undertaken. Quantitative analysis will be based on the integrated abundance from the extracted ion current profile of the primary ion (or the secondary ion if interference is observed).

The ion current profile for each peak in the sample will be integrated versus the average response factor generated from the initial multilevel calibration curve of the respective compound. For noncalibrated peaks in the sample, concentrations will be estimated by using the areas from the total ion chromatograms; the response factor for the noncalibrated compounds will be specified as 1.0. The nearest eluting internal standard in the total ion chromatogram will then be used to determine the concentration of the noncalibrated peak. Identification of the noncalibrated peak will be interpreted using reference library spectra.

Peaks are expected in the sample analyses for nontarget analytes. The laboratory will perform a library search and tentatively identify up to 10 of these unidentified peaks per sample analysis. The laboratory will supply a full validation data package for each sample, including the reference spectra and the top three spectra from the library search program.

Instrument Description

Analysis of the samples will be performed by HRGC/LRMS. The system consists of a Hewlett-Packard Model 6890 Plus GC, a Varian Archon purge-and-trap autosampler, a Tekmar Model 3000 purge-and-trap concentrator, a Hewlett-Packard 5973 MS, and a Hewlett-Packard data acquisition system running Chemserver/Target software.

C.2 Semivolatile Organic Compounds

Analysis of SVOCs collected using Method 0010 is performed using an involved two-step process. The first step, sample extraction, preparation, and clean-up, follows Method 3542. This step is used to extract the adsorbed organic compounds from the XAD-2 sorbent cartridge, from the condensate, and from the rinsate fractions of the sampling train. The resulting extract then undergoes a clean-up procedure to remove contaminants. The second step, analysis following Method 8270C, is the injection of an aliquot of the extract into the GC/MS system where the organic compounds are separated, identified, and quantified using the MS.

Preparation and Supply of Sampling Media

XAD-2 resin is a porous polymeric resin that is supplied impregnated with a bicarbonate solution to inhibit microbial growth. Both the salt solution and any residual extractable monomer and polymer species must be removed from the resin prior to use. To clean the resin, a series of washes and extractions, followed by a drying step must be performed. The first step is a water wash to remove the salt solution from the resin. The resin is placed in a beaker and rinsed with Type II water. The water is discarded along with the salt. The beaker is then filled with water again. The resin is allowed to sit in the water overnight to allow the salt in the pores of the resin to be leached. The water is drained from the beaker, and the resin is transferred to a Soxhlet or continuous extraction apparatus. The resin is then extracted for 8 hours using water. The water is drained from the resin and replaced with methanol. The resin is extracted with methanol for 22 hours. The methanol is then drained from the resin and replaced with methylene chloride. The resin is extracted with methylene chloride for 22 hours and methylene chloride is drained from the resin and replaced with fresh methylene chloride. The resin is extracted for an additional 22 hours with methylene chloride. The methylene chloride is then drained from the resin.

Following the washing and extraction sequence, the resin must be dried. The resin is transferred to a fluidized bed unit where a stream of gently heated nitrogen is passed through the resin. About 160 L of liquid nitrogen to dry a 500-g batch of extracted resin.

Once the resin has been dried, a 20-g aliquot of resin is removed, extracted according to Method 3542, and analyzed by Method 8270C (or 8290) to verify that the resin is free of interferences and background contamination. Once the resin is clean, dried, and meets method criteria, 20 g of resin will be packed into borosilicate glass thimbles. The resin will be held in place with a glass wool plug and a stainless-steel screen.

GE EER will supply the borosilicate glass thimbles containing an extra course frit to be packed with the precleaned XAD-2 resin for the Method 0010 samples.

Samples to be Collected

For each sample, one XAD-2 module, one aqueous condensate sample, a filter, and front and back half rinses will be collected and submitted to the laboratory for analysis. No samples will be collected during the drying phase of the project, and nine samples will be collected during the vitrification phase of the project. Kemron will provide the following sampling items for each run:

<u>Item</u>	<u>Number per train</u>
XAD-2 packed Module	one per sample
Filter	one per sample
Petri-dish	one per sample
1L amber glass bottle with Teflon®-lined cap	four per sample

Sample Preparation

The sample preparation procedure for the Method 0010 sampling train will follow Method 3542 and the flow chart presented in Figure C-1. The attempt has been made to maintain the lowest possible detection limits for the resulting sample extracts.

Each sample train yields five components to be extracted, concentrated, combined, and analyzed according to applicable methodologies. The five components of the sample train are as follows: (1) filter, (2) the acetone/methylene chloride rinse of the front half of the sampling train, (3) the XAD-2 sorbent trap, (4) the impinger catch and the acetone/methylene chloride rinse of the impingers, and (5) the acetone/methylene chloride rinse of the back half of the sampling train. The sample preparation procedure yields three extracts for GC/MS analysis.

Extract Number 1 includes the filter from Container 1, which is transferred to a Soxhlet apparatus. The filter is spiked with surrogate spiking standards. If particulate matter is present in the front-half rinsate (Container 2), the rinsate is filtered through another glass fiber filter. This second filter is added to the Soxhlet apparatus containing the filter from Container 1. The filters are then extracted with methylene chloride for 18 hours. The front-half of the sampling train rinsate (Container 2) is transferred to a separatory funnel. Laboratory-pure water is added to the separatory funnel. The pH of the water is adjusted to perform base/neutral/acid extractions. The methylene chloride extracts from the base/neutral/acid extractions are combined with the extract from Container 1. The combined methylene chloride extract is dried through a layer of sodium sulfate and concentrated to a final volume of 5 mL. The extract is then ready to be analyzed for semi-volatile organic compounds by EPA Method 8270C.

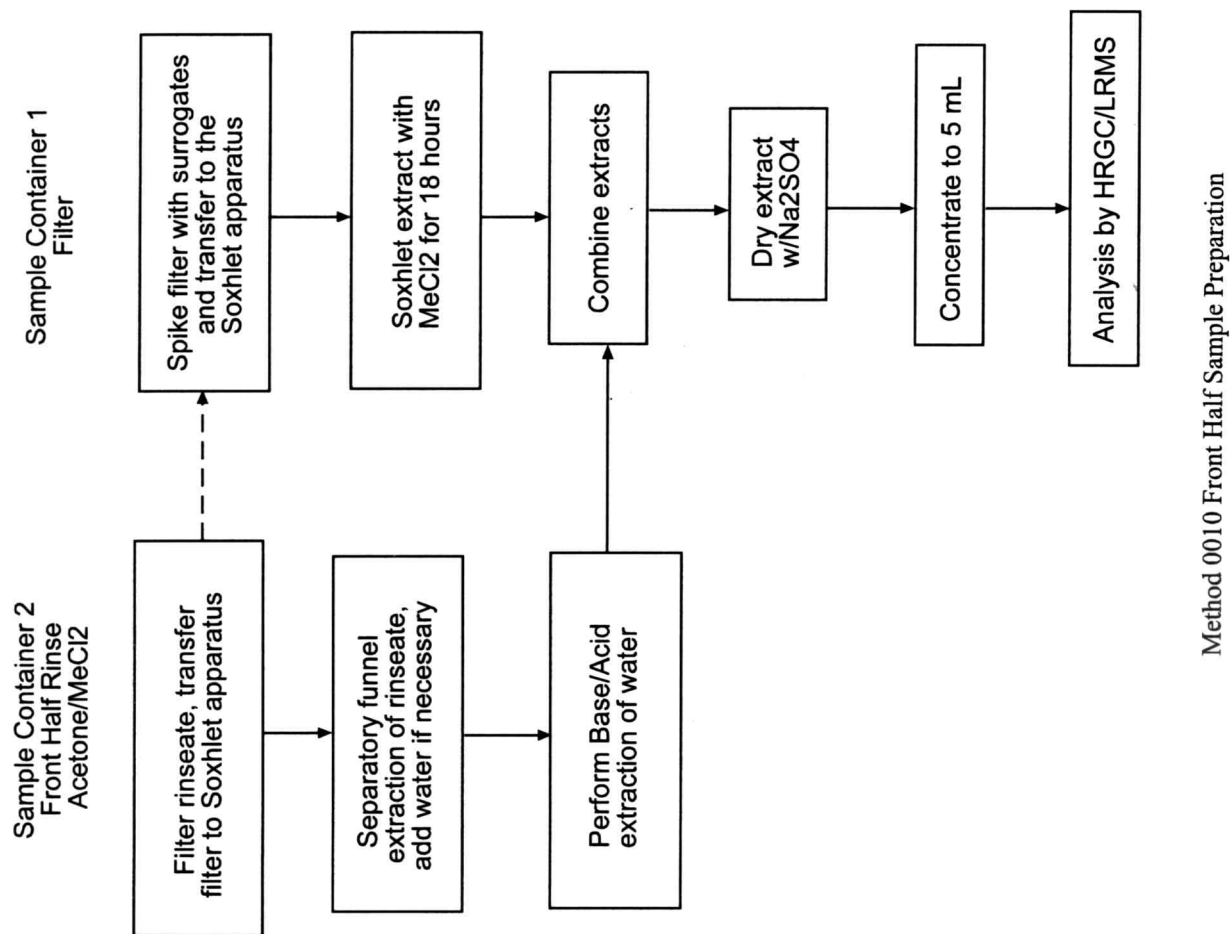
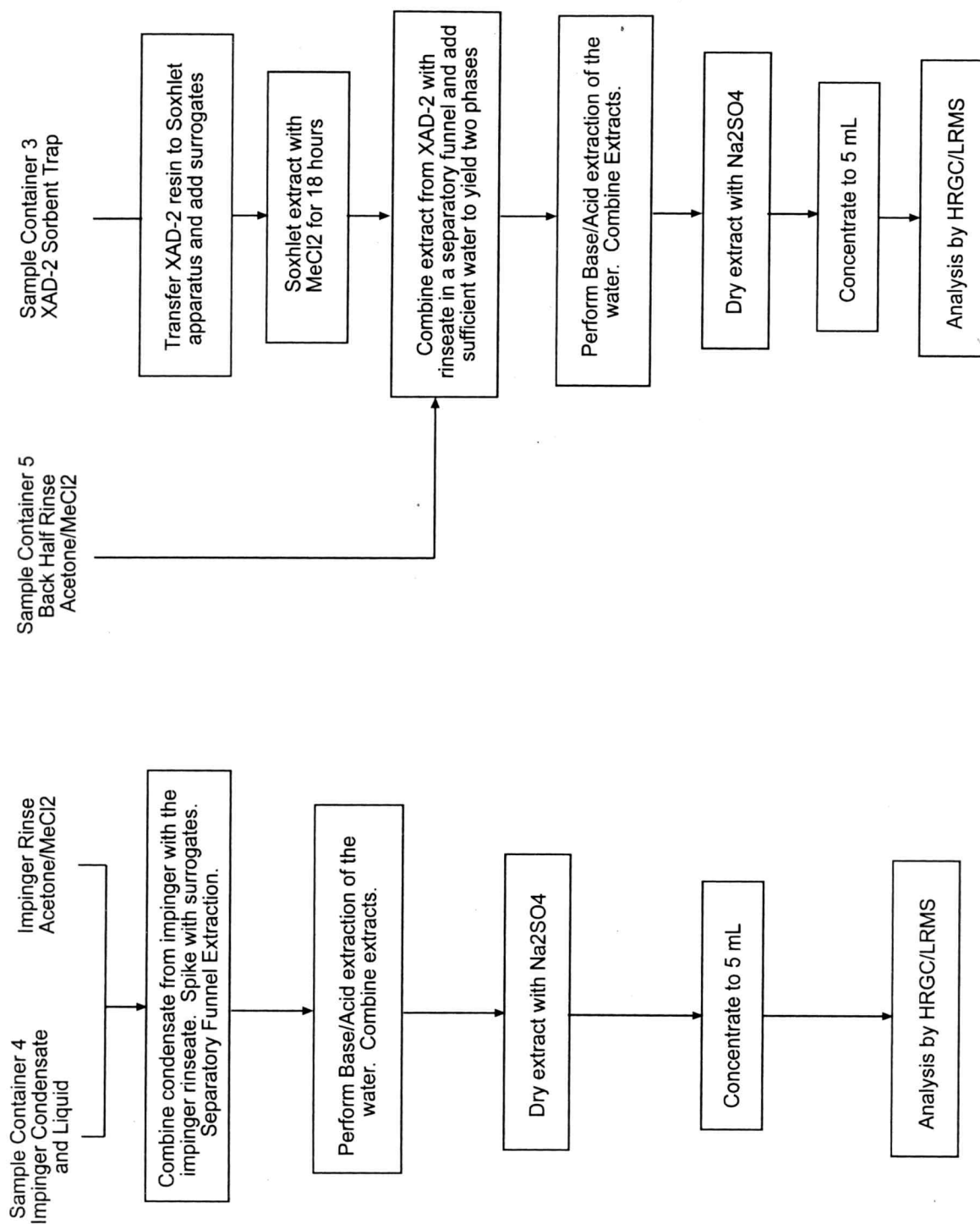


FIGURE C-1 SAMPLE PREPARATION FLOWCHART FOR SEMI-VOLATILES



Method 0010 Back Half Sample Preparation

FIGURE C-1 SAMPLE PREPARATION FLOWCHART FOR SEMI-VOLATILES (continued)

Extract Number 2 includes the XAD-2 resin from the adsorption module (Container 3), which is transferred to a Soxhlet apparatus, spiked with the surrogate spiking solution and extracted for 18 hours. The resulting extract from Container 3 is combined with the back half rinsate (Container 5) and then transferred to a separatory funnel. Sufficient laboratory-grade water, which is free of phthalates, is added to the separatory funnel to yield two separate phases. The pH is adjusted to perform base/neutral/acid extractions of the water layer. The methylene chloride extracts from the base/acid extractions are combined with the methylene chloride from the Soxhlet extraction of the XAD-2 resin. The methylene chloride is dried through a layer of sodium sulfate and concentrated to a final volume of 5 mL. The extract is then ready to be analyzed by EPA Method 8270C.

Extract Number 3 includes the condensate collected in the first impinger (Container 4) and the rinseate of the impinger, which are combined in a separatory funnel. The sample is spiked with surrogate spiking solution. Additional laboratory-grade water, which is free of phthalates, is added to the separatory funnel to yield two separate phases. The pH is adjusted to perform base/neutral/acid extractions of the water layer. The methylene chloride extracts from the base/neutral/acid extractions are combined and dried through a layer of sodium sulfate. The extract is concentrated to a final volume of 5 mL. The extract is then ready to be analyzed by EPA Method 8270C.

Table C-2 lists the components of the surrogate standard spiking solution that is added to each sample fraction as specified in the previous paragraphs.

Gas Chromatograph/Low Resolution Mass Spectrometer Calibration

At the beginning of each 12-hour analytical sequence, before any calibration standards or samples are

analyzed, 50 ng of Decafluorotriphenylphosphine (DFTPP) will be injected into the GC/MS. Mass spectral intensities for DFTPP will be generated, and these intensities will be used to verify that the mass assignments and the relative ion intensities meet the criteria of the methodology. If the criteria is met, then calibration standards may be injected, whether an initial calibration curve or a continuing calibration standard.

Calibration standards for each target compound will be generated from certified reference standards. A minimum of five calibration levels for each target compound will be generated prior to the analysis of any of the samples associated with this project are analyzed.

Kemron plans to analyze the samples collected using Method 0010 for SVOCs following Method 3540/8270C. Prior to analysis, an aliquot of the internal standard spiking solution is added to each sample extract. Table C-2 lists the components of the internal standard spiking solution.

The internal standards and surrogate standards are selected for two reasons. First, the compounds are either deuterated (contain a deuterium atom) and/or fluorinated (contain a fluorine atom), which make them highly unlikely to be found at a sufficient concentration in the environmental system under study. Second, the isotopic analogs (deuterated compounds) of the analytes of interest are expected to have similar behavior to the non-labeled compounds, thereby allowing correlation between the species.

Instrument Description

Sample extracts will be analyzed by HRGC/LRMS. The system consists of a Hewlett-Packard Model 6890 Plus GC, a Hewlett-Packard 7683 autosampler, a Hewlett-Packard 5973 MS, and a Hewlett-Packard data acquisition system running Chemserver/Target processing software. A 1-: L aliquot of extract will be injected into the GC for every reference standard and sample analysis.

TABLE C-2
STANDARDS FOR METHOD 8270C

Surrogate Standards	
2-Fluorophenol	2-Fluorobiphenyl
2,4,6-Tribromophenol	Nitrobenzene-d ₅
Phenol-d ₆	Terphenyl-d ₁₄
Internal Standards	
1,4-Dichlorobenzene-d ₄	Phenanthrene-d ₁₀
Naphthalene-d ₈	Chrysene-d ₁₂
Acenaphthene-d ₁₀	Perylene-d ₁₂

Sample Analysis

After the analytical system is calibrated, or the calibration has been verified by the analysis of the continuing calibration standard, the samples may be analyzed. An aliquot of the internal standard spiking solution (see Table C-2) is added to each sample extract. The extracts are placed into the autosampler for injection into the GC/MS system for analysis. The samples may be analyzed until the 12-hour calibration window has expired. Once expired, the calibration of the system will be verified, and another 12-hour calibration window begins. If the concentration of an analyte in a sample extract exceeds the calibration range of the instrument, the extract is diluted and reanalyzed. A proper dilution will yield a peak in the upper half of the calibration range for that compound. If the concentration of a compound in an extract is sufficient to overload the analytical column or saturate the detector signal, an instrument blank should be analyzed to verify that the system is free of contamination prior to the analysis of any additional sample extracts.

After the major components in each of the sample extracts have been identified, quantitative analyses of those compounds will be undertaken. Quantitative analysis will be based on the integrated abundance from the extracted ion current profile of the primary ion (or the secondary ion if interference is observed). The ion current profile for each peak in the sample will be integrated versus the average response factor generated from the initial multilevel calibration curve of the respective compound. For noncalibrated peaks in the sample, concentrations will be estimated by using the areas from the total ion chromatograms, and the response factor for the noncalibrated compounds will be specified as 1.0. The nearest eluting internal standard in the total ion chromatogram will then be used to determine the concentration of the noncalibrated peak. Identification of the noncalibrated peak will be interpreted using reference library spectra.

Peaks are expected in the sample analyses for nontarget analytes. The laboratory will perform a library search and tentatively identify up to 10 of these unidentified peaks per sample analysis. The laboratory will supply a full data validation package for each sample, including the reference spectra and the top three spectra from the library search program.

C.3 Polychlorinated Biphenyls and Polychlorinated Dibenzo-p-Dioxins/Dibenzofurans

PCB congeners, PCDDs, and PCDFs collected using EPA Method 23 will be analyzed using an involved two-step process. The first step (sample extraction, preparation, and clean-up) follows Method 3542. This step is used to extract the organics compounds from the sorbent cartridge, condensate, and rinseate, then cleanup the resulting extract. The second step, analysis following EPA Method 680 (PCBs) (Note: this extract will be split and one-half retained for analysis by high-resolution gas chromatography/high-resolution mass spectrometry, Method 1668, depending on the concentrations detected by Method 680) or EPA Method 8290 (PCDD/F), the injection of an aliquot of the sample extract into the GC where the organic compounds are separated, then identified and

quantified using the mass spectrometer. The samples collected during the first phase (drying step) of the project will be analyzed for PCB congeners only.

Preparation and Supply of Sampling Media

The procedure for preparing the XAD-2 resin for PCBs and dioxins and furans is identical to the procedure for SVOCs summarized previously in Section C.2.

Once the resin has been dried, a 20-g aliquot of resin is removed, extracted, and analyzed by HRGC/HRMS to verify that the resin is free of interferences and background contamination. Once the resin has met the method QA criteria, 20 g of resin will be packed into borosilicate glass thimbles. The resin will be held in place with a glass wool plug and a stainless-steel screen.

GE EER will supply Paradigm Analytical Laboratories with the borosilicate glass thimbles containing an extra course frit to be packed with the pre-cleaned XAD-2 resin. Once the thimbles are packed, the resin will be spiked with the sampling standard spiking solution.

Samples to be Collected

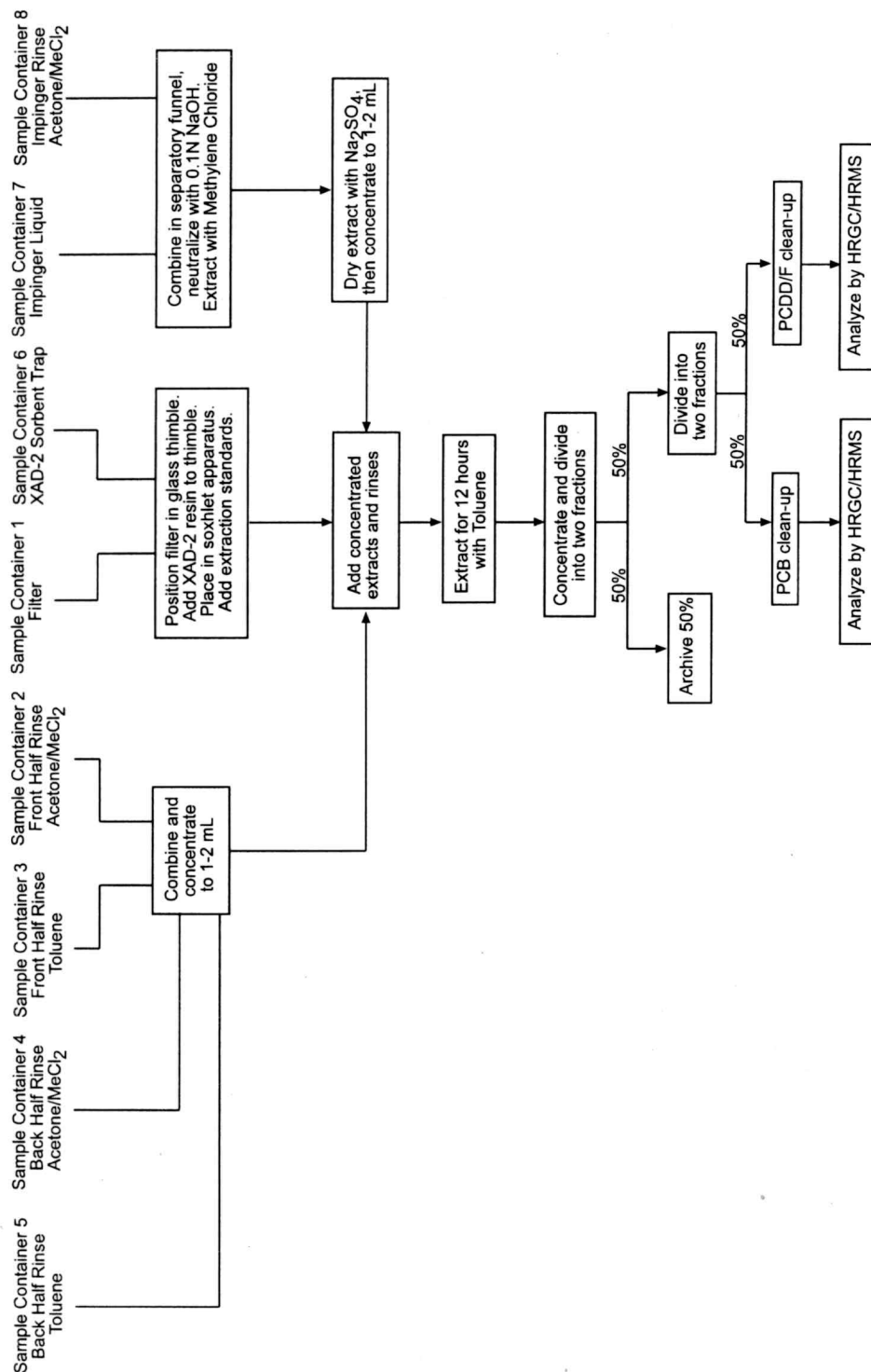
For each sample, the following will be collected and submitted to the laboratory for analysis: one XAD-2 module, one aqueous condensate sample, a filter, and front and back half rinses. The plan is to collect six samples during the drying phase of the project and 18 samples during the vitrification phase of the project. The number of samples collected may be reduced depending on the operation of the process systems. Paradigm Analytical Laboratories will provide the following sampling items for each run:

<u>Item</u>	<u>Number per train</u>
XAD-2 packed Module	1 per sample
Filter	1 per sample
Petri-dish	1 per sample
1L amber wide mouth glass bottle with Teflon®-lined cap	6 per sample

Sample Preparation

The sample preparation procedure for the Method 23 sampling train will follow the flow chart presented in Figure C-2. For the samples collected during the vitrification phase of the project, the extract will be analyzed for both PCBs and PCDD/PCDFs. The sample preparation procedure for this sampling event will be unique. The attempt has been made to maintain the lowest possible detection limits for both parameters.

Each sample train is composed of eight components to be extracted, concentrated, combined, and divided according to applicable methodologies. The eight components of the sample train are: (1) filter, (2) the acetone/methylene chloride rinse of the front half of the sampling train, (3) the toluene rinse of the front half of the sampling train, (4) the acetone/methylene chloride rinse of the back half of the sampling train, (5) the toluene rinse of the back half of the sampling train, (6) the XAD-2 sorbent trap, (7) the condensate and impinger catch, and (8) the acetone/methylene chloride rinse of the impingers of the sampling train. One extract will be prepared for analysis as described below.



Modified Method 23 Sample Presentation

FIGURE C-2 SAMPLE PREPARATION FLOWCHART FOR PCBs/PCDDs/PCDFs

The filter from Container 1 is transferred to a glass thimble. The filter is positioned so that it conforms to the inner surface of the thimble. The XAD-2 resin from Container 5 is added to the glass thimble. The surface of the XAD-2 resin is spiked with the extraction standards. The thimble is then placed into the Soxhlet apparatus.

The rinsates from Containers 2, 3, 4, and 5 are combined and concentrated to 1 to 2 mL and then added to the resin in the Soxhlet apparatus. The impinger liquids from Container 7 are combined with the impinger rinsate from container 8 in a separatory funnel. The aqueous layer is neutralized with 0.1N sodium hydroxide. The water is extracted with methylene chloride. The extract is dried with sodium sulfate, then concentrated to 1 to 2 mL. The concentrated extract is added to the resin in the Soxhlet apparatus. The filter/XAD-2 resin is then extracted with toluene for 12 hours. The resulting extract is concentrated to 1.0 mL, then split into 0.5-mL aliquots. One aliquot is archived for future analysis, if required. The other aliquot is again split into 0.25-mL aliquots. One aliquot is spiked with PCB cleanup standards and cleaned-up for PCBs prior to analysis by GC/MS. The other aliquot is cleaned-up for PCDDs and PCDFs prior to analysis by HRGC/HRMS. Table C-3 presents the extraction and cleanup standards for PCB and PCDD/PCDF samples.

High Resolution Gas Chromatograph/High Resolution Mass Spectrometer Calibration

The high resolution mass spectrometer will be tuned with perfluorokerosene (PFK) prior to each analytical period to meet the minimum required resolving power of 10,000 (10 percent valley) at mass-to-charge ratio (m/z) = 304.9824. Pentachlorobiphenyl has a m/z = 305. The resolution and exact m/z of three to five reference peaks covering the three mass ranges of interest will be monitored and recorded. The resolution must be greater than or equal to 10,000, and the deviation between the exact m/z and the theoretical m/z for each exact m/z monitored must be less than 5 ppm.

Inject the lowest calibration solution to determine ion abundance ratios, minimum levels, signal-to-noise ratios, and absolute retention times for the instrument. Operate the mass spectrometer in the mass-drift correction mode using PFK to provide the lock m/z . If the lowest calibration standard meets the QC limits of the methodology, then analyze the standards for the remaining four calibration levels. If the QC limits are not met, the mass spectrometer must be adjusted, and the analysis must be repeated until the QC requirements are met. Signal-to-noise criteria and linearity for each analyte must meet minimum criteria.

Calibration standards for each target compound will be prepared from certified reference standards. A minimum of five calibration levels for each target compound will be generated prior to the analysis of any of the samples associated with this project. Extraction standards for PCBs, PCDDs, and PCDFs are listed in Table C-3.

Paradigm Analytical Laboratories plans to analyze the samples collected using Method 23 for both PCBs using EPA Method 680 and PCDDs and PCDFs by Method 8290.

Instrument Description

Sample extracts will be analyzed by HRGC/HRMS. The system consists of a Hewlett-Packard Model 6890 Plus GC, a Leap CTC A200SW autosampler, a Micromass Autospec Ultima MS, and a OPUS MS data acquisition system. A 2-: L aliquot of extract will be injected into the GC for every reference standard and sample analysis.

TABLE C-3
Extraction Standards for PCBs, PCDDs, and PCDFs

PCB Extraction Standards	Dioxin/Furan Extraction Standards
¹³ C-3-MoCB	¹³ C ₁₂ -2,3,7,8-TCDD
¹³ C-15-DiCB	¹³ C ₁₂ -1,2,3,7,8-PeCDD
¹³ C-28-TriCB	¹³ C ₁₂ -1,2,3,6,7,8-HxCDD
¹³ C-81-TeCB	¹³ C ₁₂ -1,2,3,4,6,7,8-HpCDD
¹³ C-77-TeCB	¹³ C ₁₂ -OCDD
¹³ C-118-PeCB	¹³ C ₁₂ -2,3,7,8-TCDF
¹³ C-105-PeCB	¹³ C ₁₂ -1,2,3,7,8-PeCDF
¹³ C-126-PeCB	¹³ C ₁₂ -1,2,3,6,7,8-HxCDF
¹³ C-167-HxCB	¹³ C ₁₂ -1,2,3,4,6,7,8-HpCDF
¹³ C-156,157-HxCB	
¹³ C-169-HxCB	
¹³ C-180-HpCB	
¹³ C-189-HpCB	
¹³ C-202-OcCB	
¹³ C-208-NoCB	
¹³ C-209-DeCB	
PCB Cleanup Standards	
¹³ C-60-TeCB	
¹³ C-111-PeCB	

Notes:

MoCB- Mono-chlorinated biphenyls
DiCB- Di-chlorinated biphenyls
TriCB- Tri-chlorinated biphenyls
TeCB- Tetra-chlorinated biphenyls
PeCB- Penta-chlorinated biphenyls
HxCB- Hexa-chlorinated biphenyls
HpCB- Hepta-chlorinated biphenyls

OcCB- Octa-chlorinated biphenyls
NoCB- Nona-chlorinated biphenyls
DeCB- Deca-chlorinated biphenyls
PCB- Polychlorinated biphenyls
PCDD- Polychlorinated dibenzodioxins
PCDF- Polychlorinated dibenzofurans

C.4 Metals

Metals of interest for this project include mercury and all other species that may be volatilized during the drying process and the vitrification process. Volatilized metals will be collected using EPA Method 0060 and will be detected and quantified by the appropriate methodology, Method 6010B ICP and 7470A Cold-Vapor Atomic Absorption (CVAA). Other appropriate analytical methodologies include graphite furnace atomic absorption (GFAA). These techniques may be used in lieu of Method 6010 if lower analytical detection limits are required.

Inductively Coupled Plasma

ICP allows for the simultaneous or rapid sequential determination of many elements with detection limits comparable to or better than spectroscopy. Each sample digestate is aspirated into an argon plasma at which time element-specific emission spectra are monitored and measured by photosensitive receptors. There is the potential for interferences during the analysis of the dried sediment samples due to the high calcium content introduced by the addition of lime. However, ICP background and interelement correction can reduce the effect.

Cold-Vapor Atomic Absorption

CVAA uses a chemical reaction to reduce mercury selectively. The procedure allows mercury to be converted to its elemental state, where it is purged from the digestate and quantified using atomic absorption.

Preparation and Supply of Sampling Media

Two impinger solutions are required for collection of metals using Method 0060. A 5 percent nitric acid and 10 percent hydrogen peroxide solution (5 percent HNO_3 /10 percent H_2O_2) will be prepared before departing for the field by carefully adding 50 ml of concentrated HNO_3 to a 1,000 mL volumetric flask containing 500 mL of reagent-grade water. Next 333 mL of 30% hydrogen peroxide is slowly added to the flask. The solution is brought to volume with reagent-grade water. A 4 percent potassium permanganate and 10 percent sulfuric acid solution (4 percent KMnO_4 /10 percent H_2SO_4) will be prepared daily in the field. This solution is prepared by carefully adding 100 mL of concentrated sulfuric acid to a 1000 mL volumetric flask containing 800 mL of reagent-grade water. The solution is brought to volume with reagent-grade water. Then 40 grams of potassium permanganate (KMnO_4) crystals are dissolved into the resulting solution. The solution is then filtered through Whatman 541 filter paper to prevent the autocatalytic decomposition of the permanganate solution. The solution is stored in an amber glass bottle with a No. 70-72 hole drilled in the cap to relieve the pressure from the reaction of the permanganate with the acid.

Samples to be Collected

During the drying process, a total of four sample runs will be collected from the process for mercury and the metals chosen for analysis. During the vitrification process, a total of seven sample runs will be collected from the flue gas for the same list of metals.

Two unique halves of the sampling train are collected with each Method 0060 sample. The sample train halves are the front half (5 percent HNO_3 /10 percent H_2O_2), which will be analyzed for all volatile metal species and mercury, and the back half (4 percent KMnO_4 /10 percent H_2SO_4), which will only be analyzed for mercury. The following items are required for each run:

<u>Item</u>	<u>Number per run</u>
5 percent HNO ₃ /10 percent H ₂ O ₂ solution	200 mL
4 percent KMnO ₄ /10 percent H ₂ SO ₄ solution	200 mL
Quartz or Glass Filter	1
500-mL Nalgene [®] bottle	1
500-mL Amber glass bottle with Teflon-lined caps	3
250-mL Amber glass bottle with caps	2

Sample Preparation

The laboratory will receive a series of containers, labeled 1 through 5, for each sample train collected in the field. From these containers, three digestates will be prepared for analysis by ICP and CVAA, or just CVAA. Container 1 will contain the filter, Container 2 will contain the acetone rinse of the probe and front half of the filter housing, and Container 3 will contain the acid rinse of the probe and front half of the filter housing. These three containers will be digested, combined, and split for analysis by both ICP and CVAA. Container 4 will contain the contents of the first three impingers of the sampling train and the associated rinsates. An aliquot of the resulting solution will be removed for mercury analysis, then the remainder is digested for analysis by ICP. Container 5A contains the contents of the fourth impinger and the rinsate. Container 5B contains the contents of the fifth and sixth impingers and the rinsates. Container 5C will contain the 8N hydrogen chloride rinsate to remove any residue which may be present in the fifth and sixth impingers. The containers identified as 5A, B, and C will be digested separately and analyzed for mercury only.

Sample Analysis

Analysis of the sample digestates for all analytes except mercury will be performed on a Thermo-Jarrell Ash Supertrace ICP /purge/ spectrophotometer. The sample digestates for mercury will be analyzed on a Leeman 2000PS mercury analyzer.

After the instrument has been allowed to become thermally stable and the plasma operating conditions have been optimized, the instrument will be profiled and calibrated. The initial calibration verification (ICV), a calibration blank, and a continuing calibration verification (CCV) will be analyzed. Before each sample is analyzed, the system will be rinsed with the calibration blank solution to ensure that the system is free of contamination. After each 10 sample analyses, either an ICV or a CCV will be analyzed to verify the system is stable and operating properly. The ICV and CCV will be alternated during the analytical sequence. If the concentration of an element exceeds the calibration range of the instrument, the sample is diluted to within the calibration range and reanalyzed for that element. After all of the samples have been analyzed, the analytical sequence will be closed with the analysis of either the ICV or the CCV. Method criteria must be met for the ICV or CCV, or else the samples analyzed after the last successful ICV or CCV must be reanalyzed. The targeted metals are listed in Table C-4. The wavelengths to be monitored for each metal are listed in Table C-5.

C.5 Hydrogen Chloride and Chlorine

Samples collected during the vitrification phase of the project using EPA Method 0050, or the equivalent Method 26, will be analyzed by EPA Method 9057 (which is identical to Method 26). The chloride ion concentration will be determined in the impinger solutions using ion chromatography.

Preparation and Supply of Sampling Media

Two separate solutions are required for the sampling train. A 0.1N sulfuric acid solution is prepared for the collection of hydrogen chloride. A 0.1N sodium hydroxide solution is prepared for the collection of chlorine from the gas stream. These sampling media are prepared from reagent grade chemicals and reagent water. Both solutions will be purchased as certified solutions. Each solution will be analyzed prior to the sampling event to verify that the reagents are free of interferences and background contamination.

TABLE C-4
TARGET VOLATILE METAL SPECIES

Analyte	Sample Fraction
Antimony, Arsenic, Cadmium, Selenium, Lead, Chromium, Thallium	Front
Mercury	Front and Back

TABLE C-5
Recommended Wavelengths

Element	Wavelength (nm)	Technique
Mercury	253.7	CVAA
Antimony	206.833	ICAP
Arsenic	193.696	ICAP
Cadmium	226.502	ICAP
Selenium	196.026	ICAP
Lead	220.353	ICAP
Chromium	267.716	ICAP
Thallium	190.864	ICAP

Notes:

CVAA- Cold vapor atomic absorption

ICAP- Inductively Coupled Atomic Plasma

Samples to be Collected

During the vitrification phase of the project, GE EER will collect one Method 26 (Method 0050) train for each of the nine planned sampling runs. Each train will consist of six impingers. The first three impingers will contain 0.1N sulfuric acid, the next two impingers will contain 0.1N sodium hydroxide, and the last impinger will contain silica gel (see box below). The contents of each of the paired impingers will be combined into two separate samples.

<u>Item</u>	<u>Volume per run</u>
0.1N H ₂ SO ₄ solution	250 mL
0.1N NaOH solution	200 mL
500 mL Nalgene [®] bottles	2

Sample Preparation

The samples will be returned to the laboratory and refrigerated at $4\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$ until analysis. The volume of the bottle will be checked for leakage. The contents of the bottle will then be transferred to a 500-mL volumetric flask and brought to volume with reagent-grade water. The samples will be analyzed in duplicate after the instrument is calibrated.

Instrument Calibration

The ion chromatograph (IC) will be calibrated at a minimum of four levels at the beginning of each analytical sequence. The calibration solutions must be prepared in the solution (0.1 N sodium hydroxide or 0.1N sulfuric acid) as the samples. The sequence will be started after a stable baseline has been established by analyzing a sample of laboratory-grade water to ensure the system is free of contamination before the calibration standards are injected. If no ions are detected in the water analysis, the four calibration standards are injected. The linear correlation coefficient must be greater than or equal to 0.995 for the curve to be acceptable. After the calibration curve has been generated and after every

10 samples, a QC check sample is injected to verify the performance of the analytical system. The calculated value of the QC check sample should be within 10 percent of the certified value. Each of the calibration standards will be reinjected at the close of the analytical sequence. The calculated results for the closing analytical standards should be within 10 percent of their actual concentration.

Sample Analysis

Analysis of the sample preparations will be performed on the IC according to Method 9057. All samples will be injected in duplicate. A 25-: L aliquot is injected into the IC, and the anions are separated on an ion exchange column. The anions are detected with an electrolytic conductivity detector. A calibration curve is generated (as noted in the previous section) for each type of impinger solution (0.1N sodium hydroxide and 0.1N sulfuric acid) prior to the associated samples. Prior to the analysis of any samples, a second source QC check is analyzed to verify the calibration. This QC check sample must agree within 10 percent of the certified value. Each type of impinger catch solution is analyzed with the associated curve. Each sample is analyzed in duplicate. The results of the duplicate injections must agree within 5 percent of each other. If the duplicates do not meet this criteria, the analysis will be performed again, with all four values used to calculate the average response. A QC check will be analyzed after every 10 samples. If the QC check fails, the system will be recalibrated, and the previous 10 samples will be reanalyzed in duplicate. A matrix spike (MS) and a matrix spike duplicate (MSD) will be performed for every 20 samples per type of impinger solution. Recovery for both the MS and MSD must be between 80 and 120 percent. If the MS/MSD fail the recovery criteria, a second MS/MSD will be performed. In the event that an analyte concentration exceeds the calibration range, the sample will be diluted with the appropriate solution.

Ion Chromatograph Description

The impinger solutions will be analyzed by IC. The system consists of a Dionex 20001 IC with a conductivity detector, self-regenerating conductivity suppression, an isocratic pump, and data acquisition using TurboChrom software. A 50-: L aliquot of each sample preparation will be injected into the IC using an autosampler. A Dionex AS4A anion analytical column with AG4A guard column

Section: C.0
Revision: 2
Date: 7/24/01
Page: C-31

will be used for the analytical separation. The mobile phase for the separation will be 2.1 milliMolar (mM) sodium bicarbonate 1.2 mM sodium carbonate solution. The ion chromatograph will be calibrated for each sampling matrix. Each calibration curve will encompass a minimum of four calibration levels.